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NIST Inter-Comparison Exercise Program for Polybrominated Diphenyl Ethers (PBDEs) in Marine Sediment: Description and Results of the 2004 Inter-Comparison Exercise

Heather M. Stapleton, Jennifer M. Keller, Michele M. Schantz,
John R. Kucklick, and Stephen A. Wise

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Heather M. Stapleton, Jennifer M. Keller¹, Michele M. Schantz,
John R. Kucklick¹ and Stephen A. Wise
Analytical Chemistry Division
Chemical Science and Technology Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899-8392
and ¹Charleston, SC 29412

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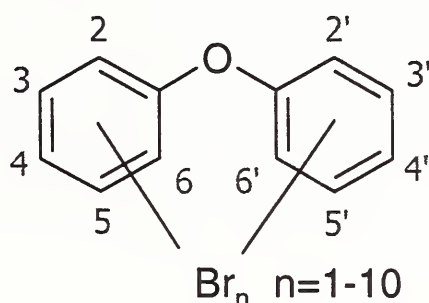
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Abstract

In support of environmental monitoring measurement programs, the National Institute of Standards and Technology (NIST) conducts inter-laboratory comparison exercises to provide one mechanism for participating laboratories/monitoring programs to evaluate the quality and comparability of their performance in measuring selected organic contaminants in environmental samples. Polybrominated diphenyl ethers (PBDEs) are brominated flame retardants that are now becoming a contaminant of emerging concern. Many studies have reported different analytical methods for measuring these contaminants in environmental samples; however, very few inter-comparison exercises have been conducted on the measurements of PBDEs in environmental samples. The one major comparison that was conducted has shown large variations in measurement values¹. This report describes the results from the NIST 2004 exercise for the determination of PBDE congeners in marine sediments. Summaries are provided with the results received from twelve participating laboratories from six different countries. A detailed analysis of the measurement variability for eight individual BDE congeners is provided. The analytical methods used by each participating laboratory are also included.

Introduction

Polybrominated diphenyl ethers (PBDEs) are flame retardant chemicals added to numerous textiles, rubbers and polymeric materials. PBDEs are now considered to be ubiquitous contaminants, and much attention has been focused on their fate and transport in the environment. Due to increased attention, many laboratories have been studying different methods to measure the suite of 209 possible isomers of PBDEs. These compounds (see Figure 1) are similar in structure to polychlorinated biphenyls (PCBs) and are labeled according to the same IUPAC labeling scheme.



Polybrominated Diphenyl Ether (PBDE)

Figure 1. Structure of polybrominated diphenyl ethers. PBDEs can have from one to ten bromine atoms substituted around the diphenyl ether backbone.

Due to increased attention and potential policy implications, the ability of laboratories to accurately determine and measure PBDEs has become important. Tools and mechanisms for the assessment of data produced by laboratories providing environmental analyses are critical because decision-making based on inaccurate results or data of unknown quality can have significant economic and health consequences. The National Institute of Standards and Technology (NIST) offers a variety of activities in support of environmental monitoring programs with the goal of improving measurements for monitoring organic contaminants. The current inter-comparison exercise was undertaken to assess the variability in PBDE measurements made by different laboratories which analyze the same reference materials using different extraction and/or detection methods. This exercise followed up on an earlier inter-comparison exercise which found large variations in the accuracy of PBDE measurements¹.

Current participants in this study represent multi-laboratory monitoring programs as well as a number of individual programs, and include federal, state/municipal, university/college, private sector, and international laboratories. In this performance based program, each participating laboratory uses its current methods for analysis of

similar materials that it would use for its program customers. The target PBDE congeners are listed in Table 1.

Sources and Preparation of Materials used in 2004 Inter-comparison Exercise

Two marine sediment Standard Reference Materials (SRMs) were distributed to each participating laboratory. These two SRMs, SRM 1941b (Organics in Marine Sediment) and SRM 1944 (New York/New Jersey Waterway Sediment) have been certified for other organic contaminants such as polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs) and organochlorine pesticides. SRM 1941b contains sediment collected from the Chesapeake Bay near the mouth of the Baltimore Harbor adjacent to the Francis Scott Key Bridge and is certified for 24 PAHs, 29 PCBs and 7 organochlorine pesticides. SRM 1944 was collected from six different sites along New York Bay and Newark Bay in 1994 and is certified for 24 PAHs, 35 PCBs, and 4 organochlorine pesticides. The sediment in both SRMs has been freeze-dried, sieved to 150 μm (SRM 1941b) or <60 μm (SRM 1944), homogenized in a cone blender, and radiation sterilized (^{60}Co) prior to bottling and analysis.

Storage and Distribution of Materials

Each bottle of marine sediment SRM was stored at room temperature in amber jars until shipped to each participating laboratory. Each participating laboratory in this inter-comparison exercise was sent one bottle each of SRMs 1941b and 1944. In the letter accompanying the shipments, participants were asked to analyze three replicate samples of each SRM in order to provide a more realistic assessment of laboratory precision. In addition each laboratory was requested to provide a brief description of their extraction, cleanup, and analytical procedures. SRMs were sent to the laboratories in August 2004. Laboratories were requested to submit results for the exercise by December 31, 2004. However, an extension was offered to several laboratories, and all data were submitted by March 30, 2005.

Evaluation of Exercise Results

Establishment of the Assigned Values

The following guidelines were used by the NIST exercise coordinators for the establishment of the exercise "Assigned Values". Each laboratory was asked to submit data from three replicate determinations of PBDEs in the SRMs. For each laboratory, the laboratory analyte mean of the three sample results (S1, S2, and S3) was calculated for each analyte. Non-numerical data were treated as follows: A mean "<value" was used when three "<values" were reported; NA (not analyzed/determined) was used for three

reported NAs; and, if the reported results were of mixed type, e.g., S1 and S2 were numerical values and S3 was reported as "<value", the two similar "types" were used to either determine the mean or to set a non-numerical descriptor.

For this exercise, the assigned value for each analyte was the geometric mean value of all reported values for each analyte, with the exception of BDE 209 in which the median value was used. Several laboratories reported BDE 209 values that were significantly outside the range of values reported by a majority of the participating laboratories when examined on both normal and log normal distribution plots of the data (see Appendices A and B). See Tables 2 and 3 for a listing of the reported values.

In instances in which the analyte concentration was below the detection limit of most participating laboratories, no exercise assigned value was calculated. In this data set, enough data were only available to establish assigned values for eight individual BDE congeners. In data sets such as this with a number of laboratories reporting results as "not detected" at various detection limits, there is no consensus as to what numerical value should be assigned to these results in the computation of grand means, etc.; e.g., "0", half Detection Limit (DL), and the DL value itself have all been used and the choice is influenced by the intended use of the particular data set.

Reported Results

Laboratories were assigned random numerical identification codes. There are two sets of results generated by NIST from the Gaithersburg, MD laboratory and the Charleston, SC laboratory. A laboratory was assigned the same code for each SRM analysis. The mean value reported by each laboratory is presented in Tables 2 and 3 in addition to the exercise assigned value and standard deviation of the assigned values for the eight BDE congeners. In this report, the triplicate results as reported by the laboratories for both SRMs are shown in Tables 4 and 5. These tables also include the laboratory's z-scores (see next section labeled "Performance Scores" for an explanation of z-scores) for the eight BDE congeners that had assigned values.

Performance Scores

The exercise coordinators recognize that different programs have different quality needs. The acceptability of the results submitted by a particular laboratory will be decided by the individual program(s) for which the particular laboratory provides data. Typically, the program will use these exercise results in conjunction with the laboratory's performance in the analysis of certified reference materials and/or control materials, and other quality assurance samples. These exercise results are exhibited in a number of ways in this report to facilitate their use by these programs in their acceptability assessments.

IUPAC guidelines² describe the use of z-scores for assessment of accuracy in intercomparison exercises such as those described in this report. This index assesses the

difference between the results of the laboratory and the exercise assigned value and can be used, with caution, to compare performance on different analytes and on different materials.

Accuracy Assessment (z-scores)

$$\text{z-score} = (\text{bias estimate})/(\text{performance criterion}) = (x-X)/\sigma$$

where x is the average of the individual laboratory results, X is the "Exercise Assigned Value" (exercise mean in this report), and σ is the target value for standard deviation.

As described in the IUPAC guidelines, the choice of σ is dependent upon data quality objectives of a particular program. It can be "fixed" and arrived at by perception, prescription, or reference to validated methodology (e.g., $\sigma = 0.20 X$, where X is the analyte concentration), or it can be an estimate of the actual variation (e.g., the calculated sample standard deviation, s , from the exercise data). The "fixed" performance criterion is more useful in the comparison of a laboratory's performance on different materials while the use of the actual variation may be more useful within a given exercise, for example, if the determination of a particular analyte is exceptionally problematic.

We have calculated and reported z-scores using a fixed performance criterion, where $\sigma = 0.20 X$. The z-scores calculated for these exercises can thus be interpreted as shown in the following examples:

z-score (20% X):

- +1 → laboratory result is 20% higher than the assigned value
- 2 → laboratory result is 40% lower than the assigned value

From a scientific point of view, IUPAC does not recommend the classification of z-scores but allows for a common classification as:

$ z \leq 2$	Satisfactory
$2 < z < 3$	Questionable
$ z \geq 3$	Unsatisfactory

Tables 4 and 5 report the z-scores for each laboratory's measurement of the eight BDE congeners for which values were assigned. These tables include a summary of the number of reported analytes that fall within each category for each laboratory.

Discussion

Laboratories were asked to quantify 34 individual PBDE congeners (see Table 1). Thirteen laboratories expressed interest in participating in this exercise, and twelve laboratories from six different countries returned results for both SRMs (see Appendix C). A majority of the laboratories supplied results for the eight most common PBDE congeners measured in environmental samples (BDE 28, BDE 47, BDE 99, BDE 100, BDE 153, BDE 154, BDE 183 and BDE 209). Exercise values were therefore established for these eight congeners, and z-scores were calculated for each laboratory as a measure of exercise comparability.

In general, data obtained for individual PBDE congeners were normally distributed (tested by the Shapiro Wilks test; see Appendix B). Exercise values were established for the data by taking the geometric mean of the data. However, results for BDE 209 (in both SRMs) were not normally distributed ($p < 0.01$; Shapiro Wilks test). Since the BDE 209 data were not normally distributed and because there were a few large outliers in the data, the BDE 209 median value (taken from reported laboratory data) was used as the exercise value for BDE 209 (see Tables 2 and 3). Two laboratories that used sonication as an extraction method were observed to report BDE 209 levels that were significantly below the exercise value for BDE 209 (see Appendix D). Two laboratories reported BDE 209 levels that were significantly above the exercise value for BDE 209 and some investigations into the extraction and quantification of BDE 209 may be warranted. (Since the first release of this document in April 2005, Laboratory 12 has discovered that the concentration of BDE 209 in their BDE 209 calibration solution was incorrect, resulting in their high reported values for BDE 209.)

The assigned values for the eight PBDE congeners ranged from $0.08 \text{ ng/g} \pm 0.09 \text{ ng/g}$ dry mass (BDE 183 in SRM 1941b) to $127 \text{ ng/g} \pm 411 \text{ ng/g}$ dry mass (BDE 209 in SRM 1944). BDE 209 was the dominant congener measured in both sediment SRMs which is similar to patterns observed in environmental sediment samples³. In general, PBDE concentrations were higher in SRM 1944 relative to SRM 1941b. The z-scores for the BDE congeners based on 20% of the exercise assigned value are included in Tables 4 and 5. A majority of the z-scores, based on 20%, for each laboratory are within ± 2 . Of the reported values for SRM 1941b, only 12% of the z-scores were >3 . Of the reported values for SRM 1944, only 9% of the z-scores were >3 . Z-scores calculated for BDE 209 among the laboratories displayed the widest variation (0.00 to 52.5).

Sulfur complexes are commonly found in sediment matrices and are very abundant in these two SRMs. Multiple techniques are used to remove sulfur from the extracts (e.g. activated copper), however, residual sulfur may still be present and cause matrix interferences upon gas chromatography with mass spectrometric (GC/MS) or with electron capture (GC/ECD) analysis. A recent paper has shown that zero valent iron and iron sulfides can reductively debrominate PBDEs, particularly BDE 209⁴. Therefore, the large relative standard deviations observed for the measurement of BDE 209 in these SRMs may be due to sulfur interferences. More work is needed to determine the role that sulfur complexes play in the extraction and measurement of PBDEs in these SRMs.

Inter-comparison exercises provide an important mechanism for assessing the comparability, accuracy, precision, and reproducibility of data being produced by the participating laboratories. Exercise materials similar in matrix, form, and analyte concentration to samples routinely analyzed by the laboratories are most useful for demonstrating the level of comparability and for revealing potential problem areas. For the determination of relatively low levels of PBDEs in these complex matrices, the levels of bias and reproducibility of many of the participating laboratories meet their current acceptability requirements; however, there is certainly room for improvement. Minimizing the among-laboratory biases so that the analytical variability is significantly less than the sampling variability should be an achievable goal.

Acknowledgments

The time and efforts of the analysts and management of the participating laboratories and the assistance of the NIST Standard Reference Materials Program for the procurement and preparation of the exercise materials are gratefully acknowledged.

Disclaimer

Certain commercial equipment, instruments, or materials are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are the best available for the purpose.

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Table 1. PBDE congeners quantified in this exercise and the number of laboratories reporting measurements above detection limits. Bold numbers indicate congeners for which a detailed examination of measurement variability was performed (see Tables 4-5).

<u>Congener</u>		<u>1944 # Labs Reporting</u>	<u>1941b # Labs Reporting</u>
BDE 15	4,4'-dibromodiphenyl ether	2	2
BDE 17	2,2',4-tribromodiphenyl ether	6	6
BDE 25	2,3',4-tribromodiphenyl ether	0	0
BDE 28	2,4,4'-tribromodiphenyl ether	7	11
BDE 30	2,4,6-tribromodiphenyl ether	0	0
BDE 33	2',3,4-tribromodiphenyl ether	0	0
BDE 47	2,2',4,4'-tetrabromodiphenyl ether	11	12
BDE 49	2,2',4,5'-tetrabromodiphenyl ether	6	7
BDE 66	2,3',4,4'-tetrabromodiphenyl ether	4	5
BDE 71	2,3',4',6-tetrabromodiphenyl ether	4	2
BDE 75	2,4,4',6-tetrabromodiphenyl ether	1	1
BDE 85	2,2',3,4,4'-pentabromodiphenyl ether	6	5
BDE 99	2,2',4,4',5-pentabromodiphenyl ether	10	11
BDE 100	2,2',4,4',6-pentabromodiphenyl ether	9	9
BDE 116	2,3,4,5,6-pentabromodiphenyl ether	1	0
BDE 118	2,3',4,4',5-pentabromodiphenyl ether	0	0
BDE 119	2,3',4,4',6-pentabromodiphenyl ether	1	1
BDE 138	2,2',3,4,4',5-hexabromodiphenyl ether	5	1
BDE 153	2,2',4,4',5,5'-hexabromodiphenyl ether	11	9
BDE 154	2,2',4,4',5,6'-hexabromodiphenyl ether	10	9
BDE 155	2,2',4,4',6,6'-hexabromodiphenyl ether	1	1
BDE 156	2,3,3',4,4',5-hexabromodiphenyl ether	0	0
BDE 181	2,2',3,4,4',5,6-heptabromodiphenyl ether	1	1
BDE 183	2,2',3,4,4',5',6-heptabromodiphenyl ether	11	6
BDE 190	2,3,3',4,4',5,6-heptabromodiphenyl ether	5	1
BDE 191	2,3,3',4,4',5',6-heptabromodiphenyl ether	0	0
BDE 196	2,2',3,3',4,4',5,6'-octabromodiphenyl ether	2	1
BDE 197	2,2',3,3',4,4',6,6'-octabromodiphenyl ether	2	1
BDE 203	2,2',3,4,4',5,5',6-octabromodiphenyl ether	5	1
BDE 205	2,3,3',4,4',5,5',6-octabromodiphenyl ether	1	1
BDE 206	2,2',3,3',4,4',5,5',6-nonabromodiphenyl ether	6	4
BDE 207	2,2',3,3',4,4',5,6,6'-nonabromodiphenyl ether	3	2
BDE 208	2,2',3,3',4,5,5',6,6'-nonabromodiphenyl ether	2	1
BDE 209	2,2',3,3',4,4',5,5',6,6'-decabromodiphenyl ether	11	10

Table 2. Laboratory means of three replicates and exercise mean and standard deviation for SRM 1941b. (Concentration is in ng/g dry mass.)

<u>Congener</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>	Geometric		Exercise
													Mean	Std. Dev.	
BDE 15	NR	NR	NR	0.25	0.11	NR	NR	NR	NR	<0.2	NR	NR			
BDE 17	0.12	0.16	NR	0.21	0.11	NR	<0.04	NR	NR	<0.2	0.06	0.15			
BDE 25	NR	NR	NR	NR	NR	NR	<0.03	NR	NR	<0.2	NR	NR			
BDE 28-33	0.20	0.20	0.17	0.21	0.13	NR	0.11	0.12	0.35	0.22	0.13	0.23	0.18	0.07	
BDE 30	NR	<0.1	NR	<0.003	NR	NR	<0.04	NR	NR	<0.2	NR	NR			
BDE 33	NR	NR	NR	NR	NR	NR	NR	NR	NR	<0.2	NR	NR			
BDE 47	1.82	1.47	1.41	1.47	1.47	1.46	0.85	1.64	2.72	1.68	0.76	2.08	1.48	0.51	
BDE 49	NR	0.32	NR	0.21	0.25	0.24	0.15	0.33	NR	0.19	NR	NR			
BDE 66	0.06	<0.1	NR	0.06	0.05	NR	<0.05	<0.1	0.10	<0.2	ND	0.05			
BDE 71	NR	<0.1	NR	0.02	0.02	NR	<0.03	<0.1	NR	<0.2	ND	ND			
BDE 75	NR	<0.1	NR	0.01	NR	NR	<0.03	<0.1	NR	<0.2	NR	NR			
BDE 85	0.05	0.19	NR	0.02	0.02	NR	<0.03	0.22	ND	<0.2	ND	ND			
BDE 99	0.80	0.55	0.55	0.52	0.64	0.63	0.46	0.42	1.08	0.59	ND	0.82	0.62	0.19	
BDE 100	0.17	0.14	NR	0.15	0.15	NR	0.11	0.10	0.29	0.12	ND	0.22	0.15	0.06	
BDE 116	NR	<0.1	NR	<0.004	NR	NR	<0.07	<0.1	NR	<0.2	NR	NR			
BDE 118	NR	<0.1	NR	NR	NR	NR	NR	NR	NR	<0.2	NR	NR			
BDE 119	NR	<0.1	NR	0.02	<0.002	NR	<0.03	NR	NR	<0.2	NR	ND			
BDE 138	ND	<0.1	NR	0.01	<0.01	NR	<0.03	<0.1	NR	<0.2	ND	ND			
BDE 153	0.11	<0.1	0.07	0.08	0.08	NR	0.06	0.17	0.12	0.07	ND	0.08	0.09	0.04	
BDE 154	0.13	<0.1	0.07	0.09	0.09	NR	0.06	0.13	0.08	0.07	ND	0.09	0.09	0.02	
BDE 155	NR	<0.1	NR	0.02	NR	NR	<0.03	NR	NR	<0.1	NR	ND			

Table 2. Laboratory means of three replicates and exercise mean and standard deviation for SRM 1941b. (Concentration is in ng/g dry mass.) (continued)

<u>Congener</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>	Geometric		Exercise
													Mean	Std. Dev.	
BDE 156	NR	<0.1	NR	NR	NR	NR	<0.03	NR	NR	<0.2	NR	NR			
BDE 181	NR	<0.2	NR	0.06	NR	NR	<0.04	NR	NR	<0.2	NR	NR			
BDE 183	0.05	<0.2	<0.04	0.04	0.06	NR	<0.04	NR	0.10	0.05	ND	0.05	0.05	0.02	
BDE 190	ND	<0.2	NR	0.14	NR	NR	<0.03	<1	NR	<0.2	ND	ND			
BDE 191	NR	<0.2	NR	NR	NR	NR	<0.03	NR	NR	<0.2	NR	ND			
BDE 196	NR	0.38	NR	NR	NR	NR	NR	NR	NR	<0.4	NR	ND			
BDE 197	NR	0.26	NR	NR	NR	NR	NR	NR	NR	<0.4	NR	ND			
BDE 203	NR	<0.3	NR	0.33	NR	NR	<0.22	NR	NR	<0.4	NR	ND			
BDE 205	NR	<0.3	NR	NR	NR	NR	<0.03	NR	NR	<0.4	NR	ND			
BDE 206	NR	2.76	NR	NR	NR	NR	0.62	NR	NR	2.10	NR	ND			
BDE 207	NR	1.77	NR	NR	NR	NR	1.32	NR	NR	NR	NR	0.57			
BDE 208	NR	NR	NR	NR	NR	NR	0.48	NR	NR	NR	NR	ND			
BDE 209	8.05	63.67	22.58	22.73	NR	22.93	21.40	25.84	25.33	25.29	NR	145.33 ^a	24.11 ^b		14.97

NR – indicates not reported.

ND - indicates not detected.

^a Since the first release of this document, Laboratory 12 has discovered that their BDE 209 calibration solution was incorrectly calibrated by a factor of 5. Their recent reported average value for BDE 209 is 28.8 ng/g.

^b This is the median value which was used as the exercise value because the data set was not log normally distributed.

Table 3. Laboratory means of three replicates and exercise mean and standard deviation for SRM 1944. (Concentration is in ng/g dry mass.)

<u>Congener</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>	Geometric	Exercise
													<u>Mean</u>	<u>Std. Dev.</u>
BDE 15	NR	NR	NR	0.97	NR	NR	NR	NR	NR	<0.2	NR	NR		
BDE 17	0.65	1.32	NR	0.93	0.44	NR	NR	NR	NR	<0.2	0.40	0.39		
BDE 25	NR	NR	NR	NR	NR	NR	NR	NR	NR	<0.2	NR	NR		
BDE 28-33	ND	0.20	0.13	0.30	0.16	NR	NR	<0.1	0.79	<0.2	0.51	0.17	0.26	0.24
BDE 30	NR	<0.1	NR	<0.02	NR	NR	NR	NR	NR	<0.2	NR	NR		
BDE 33	NR	NR	NR	NR	NR	NR	NR	NR	NR	<0.2	NR	NR		
BDE 47	2.18	1.84	1.01	1.62	1.39	1.62	NR	1.62	2.19	2.30	1.37	1.32	1.63	0.41
BDE 49	NR	1.74	NR	0.98	0.99	0.75	NR	1.32	NR	2.21	NR	NR		
BDE 66	ND	0.21	NR	0.23	0.08	NR	NR	<0.1	ND	<0.2	ND	0.07		
BDE 71	NR	<0.1	NR	0.18	0.13	NR	NR	0.17	NR	<0.2	0.59	NR		
BDE 75	NR	<0.1	NR	0.06	NR	NR	NR	NR	NR	<0.2	NR	NR		
BDE 85	0.22	0.47	NR	0.13	0.10	NR	NR	0.34	ND	<0.2	ND	0.12		
BDE 99	2.37	1.88	1.41	2.16	1.77	NR	NR	1.39	1.64	2.22	1.49	1.92	1.80	0.35
BDE 100	0.53	0.41	NR	0.66	0.56	NR	NR	0.32	0.51	0.50	0.30	0.47	0.46	0.12
BDE 116	NR	<0.1	NR	0.12	NR	NR	NR	<0.1	NR	<0.2	NR	NR		
BDE 118	NR	<0.1	NR	NR	NR	NR	NR	NR	NR	<0.2	NR	NR		
BDE 119	NR	<0.1	NR	0.39	<0.01	NR	NR	NR	NR	<0.2	NR	ND		
BDE 138	0.48	0.78	NR	0.83	0.88	NR	NR	NR	NR	NR	0.47	0.46		
BDE 153	6.67	7.15	6.71	8.22	7.20	5.67	NR	8.76	5.85	5.52	4.11	7.38	6.53	1.32
BDE 154	1.16	1.03	1.26	2.69	0.96	NR	NR	1.01	1.19	2.00	0.71	1.35	1.24	0.58
BDE 155	NR	<0.2	NR	0.32	NR	NR	NR	NR	NR	<0.2	NR	NR		

Table 3. Laboratory means of three replicates and exercise mean and standard deviation for SRM 1944. (Concentration is in ng/g dry mass.) (continued)

<u>Congener</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>	Geometric Mean	Exercise Std. Dev.
BDE 156	NR	<0.2	NR	NR	NR	NR	NR	NR	NR	<0.2	NR	NR		
BDE 181	NR	<0.3	NR	0.43	NR	NR	NR	NR	NR	<0.2	NR	NR		
BDE 183	34.91	28.78	32.57	36.97	36.91	21.77	NR	48.59	31.40	29.79	21.40	40.84	32.20	7.94
BDE 190	1.92	3.80	NR	4.68	NR	NR	NR	<1	NR	NR	1.43	3.31		
BDE 191	NR	<0.3	NR	NR	NR	NR	NR	NR	NR	<0.2	NR	NR		
BDE 196	NR	27.47	NR	NR	NR	NR	NR	NR	NR	NR	NR	19.40		
BDE 197	NR	16.78	NR	NR	NR	NR	NR	NR	NR	NR	NR	9.79		
BDE 203	NR	7.96	NR	6.99	NR	26.13	NR	NR	NR	9.54	NR	6.68		
BDE 205	NR	1.52	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR		
BDE 206	NR	13.84	NR	NR	NR	5.70	2.51	4.33	NR	11.02	NR	9.27		
BDE 207	NR	27.15	NR	NR	NR	NR	37.63	30.72	NR	NR	NR	36.20		
BDE 208	NR	NR	NR	NR	NR	NR	2.38	NR	NR	NR	NR	1.96		
BDE 209	34.83	127.53	134.10	124.33	NR	91.50	142.50	131.61	342.00	119.70	44.40	1467 ^a	127.53 ^b	83.75

NR – indicates not reported.

ND – indicates not detected.

^a Since the first release of this document, Laboratory 12 has discovered that their BDE 209 calibration solution was incorrectly calibrated by a factor of 5. Their recent reported average value for BDE 209 is 133.3 ng/g.

^b This is the median value which was used as the exercise value because the data set was not log normally distributed.

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 1							
Congener	1941b A	1941b B	1941b C	Ave.	SD	Exercise Value	z-score
BDE 15							
BDE 17	0.13	0.12	0.12	0.12	0.01		
BDE 25							
BDE 28-33	0.22	0.19	0.18	0.20	0.02	0.18	0.46
BDE 30							
BDE 33							
BDE 47	1.95	1.75	1.77	1.82	0.11	1.48	1.16
BDE 49							
BDE 66	0.08	0.06	0.05	0.06	0.02		
BDE 71	IS	IS	IS				
BDE 75							
BDE 85	0.04	0.04	0.06	0.05	0.01		
BDE 99	0.86	0.76	0.78	0.80	0.05	0.62	1.44
BDE 100	0.18	0.16	0.17	0.17	0.01	0.15	0.68
BDE 116							
BDE 118							
BDE 119							
BDE 138	ND	ND	ND				
BDE 153	0.12	0.12	0.10	0.11	0.01	0.09	1.31
BDE 154	0.16	0.11	0.13	0.13	0.02	0.09	2.31
BDE 155							
BDE 156							
BDE 181							
BDE 183	ND	0.03	0.07	0.05	0.03	0.08	-2.00
BDE 190	ND	ND	ND				
BDE 191							
BDE 196							
BDE 197							
BDE 203							
BDE 205							
BDE 206							
BDE 207							
BDE 208							
BDE 209	8.38	7.65	8.13	8.05	0.37	24.11	-3.33

Category	z (20%)
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Instrument Used: HRGC/MS (NCI)

GC Column:

PBDEs:	25 m HP-5MS
BDE 209:	15 m HP-1

<2	5
2 to 3	2
>3	1

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 2							
Congener	1941b A	1941b B	1941b C	Ave.	SD	Exercise Value	z-score
BDE 15	NA	NA	NA				
BDE 17	0.15	0.17	0.17	0.16	0.01		
BDE 25	other	other	other				
BDE 28-33	0.20	0.21	0.18	0.20	0.02	0.18	0.46
BDE 30	<0.1	<0.1	<0.1				
BDE 33	other	other	other				
BDE 47	1.53	1.45	1.42	1.47	0.06	1.48	-0.05
BDE 49	0.32	0.33	0.32	0.32	0.01		
BDE 66	<0.1	<0.1	<0.1				
BDE 71	<0.1	<0.1	<0.1				
BDE 75	<0.1	<0.1	<0.1				
BDE 85	0.20	0.21	0.17	0.19	0.02		
BDE 99	0.65	0.51	0.50	0.55	0.08	0.62	-0.54
BDE 100	0.14	0.13	0.14	0.14	0.01	0.15	-0.44
BDE 116	<0.1	<0.1	<0.1				
BDE 118	<0.1	<0.1	<0.1				
BDE 119	<0.1	<0.1	<0.1				
BDE 138	<0.1	<0.1	<0.1				
BDE 153	<0.1	<0.1	<0.1			0.09	
BDE 154	<0.1	<0.1	<0.1			0.09	
BDE 155	<0.1	<0.1	<0.1				
BDE 156	<0.1	<0.1	<0.1				
BDE 181	<0.2	<0.2	<0.2				
BDE 183	<0.2	<0.2	<0.2			0.08	
BDE 190	<0.2	<0.2	<0.2				
BDE 191	<0.2	<0.2	<0.2				
BDE 196	0.38	0.37	0.38	0.38	0.01		
BDE 197	0.25	0.27	0.26	0.26	0.01		
BDE 203	<0.3	<0.3	<0.3				
BDE 205	<0.3	<0.3	<0.3				
BDE 206	2.93	2.61	2.75	2.76	0.16		
BDE 207	1.73	1.56	2.03	1.77	0.24		
BDE 208	NA	NA	NA				
BDE 209	69.30	62.00	59.70	63.67	5.01	24.11	8.20

Category **z (20%)**

Instrument Used: GC/MS (NCI)

<2 4

GC Column:

2 to 3 0

PBDEs: 25 m HT-8

>3 1

BDE 209: 12 m AT-5

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 3							
<u>Congener</u>	<u>1941b A</u>	<u>1941b B</u>	<u>1941b C</u>	<u>Ave.</u>	<u>SD</u>	<u>Exercise Value</u>	<u>z-score</u>
BDE 15	NA	NA	NA				
BDE 17	NA	NA	NA				
BDE 25	NA	NA	NA				
BDE 28-33	0.16	0.17	0.19	0.17	0.02	0.18	-0.24
BDE 30	NA	NA	NA				
BDE 33	NA	NA	NA				
BDE 47	1.32	1.44	1.46	1.41	0.08	1.48	-0.25
BDE 49	NA	NA	NA				
BDE 66	NA	NA	NA				
BDE 71	NA	NA	NA				
BDE 75	NA	NA	NA				
BDE 85	NA	NA	NA				
BDE 99	0.50	0.54	0.62	0.55	0.06	0.62	-0.54
BDE 100	NA	NA	NA			0.15	
BDE 116	NA	NA	NA				
BDE 118	NA	NA	NA				
BDE 119	NA	NA	NA				
BDE 138	NA	NA	NA				
BDE 153	0.05	0.07	0.08	0.07	0.01	0.09	-1.29
BDE 154	0.06	0.08	0.07	0.07	0.01	0.09	-1.15
BDE 155	NA	NA	NA				
BDE 156	NA	NA	NA				
BDE 181	NA	NA	NA				
BDE 183	<0.04	<0.04	<0.04			0.08	
BDE 190	NA	NA	NA				
BDE 191	NA	NA	NA				
BDE 196	NA	NA	NA				
BDE 197	NA	NA	NA				
BDE 203	NA	NA	NA				
BDE 205	NA	NA	NA				
BDE 206	NA	NA	NA				
BDE 207	NA	NA	NA				
BDE 208	NA	NA	NA				
BDE 209	20.82	23.02	23.89	22.58	1.58	24.11	-0.32

<u>Category</u>	<u>z (20%)</u>
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Instrument Used: GC/EI-MS (PBDEs); GC/ECD (BDE 209)
 GC Column: PBDEs: 30 m HP-5MS
 BDE 209: 15 m DB-5

<2	6
2 to 3	0
>3	0

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 4						Exercise	
Congener	1941b A	1941b B	1941b C	Ave.	SD	Value	z-score
BDE 15	0.24	0.25	0.25	0.25	0.01		
BDE 17	0.20	0.21	0.22	0.21	0.01		
BDE 25	other	other	other				
BDE 28-33	0.19	0.20	0.24	0.21	0.02	0.18	0.88
BDE 30	<0.003	<0.002	<0.002				
BDE 33	other	other	other				
BDE 47	1.50	1.54	1.38	1.47	0.08	1.48	-0.02
BDE 49	0.21	0.20	0.21	0.21	0.00		
BDE 66	0.05	0.06	0.06	0.06	0.00		
BDE 71	0.02	0.03	0.03	0.02	0.00		
BDE 75	0.01	0.01	0.01	0.01	0.00		
BDE 85	0.02	0.02	0.02	0.02	0.00		
BDE 99	0.54	0.54	0.49	0.52	0.03	0.62	-0.79
BDE 100	0.15	0.15	0.15	0.15	0.00	0.15	0.03
BDE 116	<0.004	<0.004	<0.004				
BDE 118	N/A	N/A	N/A				
BDE 119			<0.04				
BDE 138	0.01	0.02	0.02	0.01	0.00		
BDE 153	0.08	0.09	0.09	0.08	0.01	0.09	-0.35
BDE 154	0.08	0.09	0.10	0.09	0.01	0.09	0.04
BDE 155	0.01	0.02	0.02	0.02	0.00		
BDE 156	N/A	N/A	N/A				
BDE 181	0.05	0.06	0.07	0.06	0.01		
BDE 183			0.04	0.04		0.05	-1.00
BDE 190			<0.01				
BDE 191	N/A	N/A	N/A				
BDE 196	N/A	N/A	N/A				
BDE 197	N/A	N/A	N/A				
BDE 203			0.04				
BDE 205	N/A	N/A	N/A				
BDE 206			0.58				
BDE 207			0.26				
BDE 208	N/A	N/A	N/A				
BDE 209	25.20	24.00	19.00	22.73	3.29	24.11	-0.29

Category z (20%)

Instrument Used: GC/HRMS

<2 8

GC Column:

2 to 3 0

PBDEs 30 m DB-5 HT

>3 0

BDE 209: 30 m DB-5 HT

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 5						Exercise	
<u>Congener</u>	<u>1941b A</u>	<u>1941b B</u>	<u>1941b C</u>	<u>Ave.</u>	<u>SD</u>	<u>Value</u>	<u>z-score</u>
BDE 15	0.10	0.13	0.11	0.11	0.01		
BDE 17	0.10	0.14	0.11	0.11	0.02		
BDE 25	NA	NA	NA				
BDE 28-33	0.11	0.14	0.13	0.13	0.01	0.18	-1.47
BDE 30	NA	NA	NA				
BDE 33	NA	NA	NA				
BDE 47	1.43	1.46	1.52	1.47	0.04	1.48	-0.04
BDE 49	0.26	0.24	0.25	0.25	0.01		
BDE 66	0.05	0.05	0.05	0.05	0.00		
BDE 71	0.02	0.02	0.02	0.02	0.00		
BDE 75	NA	NA	NA				
BDE 85	0.02	0.02	0.02	0.02	0.00		
BDE 99	0.61	0.65	0.65	0.64	0.02	0.62	0.15
BDE 100	0.18	0.14	0.14	0.15	0.02	0.15	-0.01
BDE 116	NA	NA	NA				
BDE 118	NA	NA	NA				
BDE 119	<0.004	<0.002	<0.002				
BDE 138	<0.010	<0.004	<0.004				
BDE 153	0.08	0.08	0.08	0.08	0.00	0.09	-0.48
BDE 154	0.13	0.07	0.07	0.09	0.03	0.09	0.15
BDE 155	NA	NA	NA				
BDE 156	NA	NA	NA				
BDE 181	NA	NA	NA				
BDE 183	0.10	0.03	0.04	0.06	0.03	0.08	-1.56
BDE 190	NA	NA	NA				
BDE 191	NA	NA	NA				
BDE 196	NA	NA	NA				
BDE 197	NA	NA	NA				
BDE 203	NA	NA	NA				
BDE 205	NA	NA	NA				
BDE 206	NA	NA	NA				
BDE 207	NA	NA	NA				
BDE 208	NA	NA	NA				
BDE 209	NA	NA	NA			24.11	-5.00
						<u>Category</u>	<u>z (20%)</u>
Instrument Used:		GC/HRMS				<2	7
GC Column:		30 m HP-5 MS				2 to 3	0
						>3	0

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 6							
Congener	1941b A	1941b B	1941b C	Ave.	SD	Exercise Value	z-score
BDE 15							
BDE 17							
BDE 25							
BDE 28-33						0.18	
BDE 30							
BDE 33							
BDE 47	1.47	1.52	1.40	1.46	0.06	1.48	-0.06
BDE 49	0.26	0.20	0.26	0.24	0.03		
BDE 66							
BDE 71							
BDE 75							
BDE 85							
BDE 99	0.70	0.47	0.72	0.63	0.14	0.62	0.07
BDE 100						0.15	
BDE 116							
BDE 118							
BDE 119							
BDE 138							
BDE 153						0.09	
BDE 154						0.09	
BDE 155							
BDE 156							
BDE 181							
BDE 183						0.08	
BDE 190							
BDE 191							
BDE 196							
BDE 197							
BDE 203							
BDE 205							
BDE 206							
BDE 207							
BDE 208							
BDE 209	22.80	21.90	24.10	22.93	1.11	24.11	-0.24

Category	z (20%)
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Instrument Used: GC/MS -EI: GC/NCI-MS (BDE 209)

GC Column:

PBDEs:	30 m DB-5MS
BDE 209:	15 m DB-5MS

<2	3
2 to 3	0
>3	0

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 7				Exercise			
<u>Congener</u>	<u>1941b A</u>	<u>1941b B</u>	<u>1941b C</u>	<u>Ave.</u>	<u>SD</u>	<u>Value</u>	<u>z-score</u>
BDE 15	NA	NA	NA				
BDE 17	<0.04	<0.04	<0.04				
BDE 25	<0.03	<0.03	<0.03				
BDE 28-33	0.10	0.13	0.11	0.11	0.02	0.18	-1.85
BDE 30	<0.04	<0.04	<0.04				
BDE 33	other	other	other				
BDE 47	0.81	0.94	0.81	0.85	0.08	1.48	-2.12
BDE 49	0.17	0.14	0.13	0.15	0.02		
BDE 66	<0.05	<0.05	<0.05				
BDE 71	<0.03	<0.03	<0.03				
BDE 75	<0.03	<0.03	<0.03				
BDE 85	<0.03	<0.03	<0.03				
BDE 99	0.55	0.44	0.40	0.46	0.08	0.62	-1.26
BDE 100	0.11	0.13	0.10	0.11	0.02	0.15	-1.22
BDE 116	<0.07	<0.07	<0.07				
BDE 118	NA	NA	NA				
BDE 119	<0.03	<0.03	<0.03				
BDE 138	<0.03	<0.03	<0.03				
BDE 153	0.06	0.06	0.06	0.06	0.00	0.09	-1.67
BDE 154	0.06	0.07	0.06	0.06	0.01	0.09	-1.48
BDE 155	<0.03	<0.03	<0.03				
BDE 156	<0.03	<0.03	<0.03				
BDE 181	<0.04	<0.04	<0.04				
BDE 183	<0.04	<0.04	<0.04			0.08	
BDE 190	<0.03	<0.03	<0.03				
BDE 191	<0.03	<0.03	<0.03				
BDE 196	NA	NA	NA				
BDE 197	NA	NA	NA				
BDE 203	<0.22	<0.21	<0.22				
BDE 205	<0.03	<0.03	<0.03				
BDE 206	0.43	0.87	0.56	0.62	0.23		
BDE 207	1.11	1.71	1.14	1.32	0.34		
BDE 208	0.40	0.63	0.40	0.48	0.13		
BDE 209	20.60	22.50	21.10	21.40	0.98	24.11	-0.56

<u>Category</u>	<u>z (20%)</u>
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Instrument Used: GC/NCI-MS
 GC Column

PBDEs: 20 m DB-1MS
 BDE 209: 20 m DB-1MS

<2	6
2 to 3	1
>3	0

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 8					Exercise		
<u>Congener</u>	<u>1941b A</u>	<u>1941b B</u>	<u>1941b C</u>	<u>Ave.</u>	<u>SD</u>	<u>Value</u>	<u>z-score</u>
BDE 15	NA	NA					
BDE 17	NA	NA					
BDE 25	NA	NA					
BDE 28-33	0.11	0.12		0.12	0.01	0.18	-1.79
BDE 30	NA	NA					
BDE 33	NA	NA					
BDE 47	1.64	1.63		1.64	0.01	1.48	0.52
BDE 49	0.34	0.32		0.33	0.01		
BDE 66	< 0,1	< 0,1					
BDE 71	< 0,1	< 0,1					
BDE 75	< 0,1	< 0,1					
BDE 85	0.24	0.21		0.22	0.02		
BDE 99	0.43	0.41		0.42	0.01	0.62	-1.62
BDE 100	0.10	<0,1		0.10		0.15	-1.73
BDE 116	< 0,1	< 0,1					
BDE 118	NA	NA					
BDE 119	NA	NA					
BDE 138	< 0,1	< 0,1					
BDE 153	0.19	0.16		0.17	0.02	0.09	4.53
BDE 154	0.15	0.11		0.13	0.02	0.09	2.28
BDE 155	NA	NA					
BDE 156	NA	NA					
BDE 181	IS	IS					
BDE 183	Other	Other				0.08	
BDE 190	<1	<1					
BDE 191	NA	NA					
BDE 196	Other	Other					
BDE 197	Other	Other					
BDE 203	Other	Other					
BDE 205	Other	Other					
BDE 206	Other	Other					
BDE 207	Other	Other					
BDE 208	NA	NA					
BDE 209	29.36	22.33		25.84	4.97	24.11	0.36

<u>Category</u>	<u>z (20%)</u>
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Instrument Used: GC/NCI-MS

GC Column:

PBDEs:	30 m RTx_CLPesticides
BDE 209:	30 m RTx_CLPesticides

<2	5
2 to 3	1
>3	1

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 9				Exercise			
Congener	1941b A	1941b B	1941b C	Ave.	SD	Value	z-score
BDE 15	NA	NA	NA				
BDE 17	NA	NA	NA				
BDE 25	NA	NA	NA				
BDE 28-33	0.38	0.33	0.34	0.35	0.03	0.18	4.71
BDE 30	NA	NA	NA				
BDE 33	NA	NA	NA				
BDE 47	2.84	2.79	2.53	2.72	0.17	1.48	4.19
BDE 49	NA	NA	NA				
BDE 66	0.09	0.10	0.10	0.10	0.01		
BDE 71	NA	NA	NA				
BDE 75	NA	NA	NA				
BDE 85	DL	DL	DL				
BDE 99	0.86	1.31	1.08	1.08	0.22	0.62	3.75
BDE 100	0.21	0.34	0.32	0.29	0.07	0.15	4.58
BDE 116	NA	NA	NA				
BDE 118	NA	NA	NA				
BDE 119	NA	NA	NA				
BDE 138	NA	NA	NA				
BDE 153	0.10	0.11	0.17	0.12	0.04	0.09	1.91
BDE 154	0.09	0.09	0.08	0.08	0.01	0.09	-0.31
BDE 155	NA	NA	NA				
BDE 156	NA	NA	NA				
BDE 181	NA	NA	NA				
BDE 183	0.09	0.11	0.11	0.10	0.01	0.08	
BDE 190	NA	NA	NA				
BDE 191	NA	NA	NA				
BDE 196	NA	NA	NA				
BDE 197	NA	NA	NA				
BDE 203	NA	NA	NA				
BDE 205	NA	NA	NA				
BDE 206	NA	NA	NA				
BDE 207	NA	NA	NA				
BDE 208	NA	NA	NA				
BDE 209	15.00	27.00	34.00	25.33	9.61	24.11	0.25

Category	z (20%)
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Instrument Used: GC/EI-MS (PBDEs); GC/NCI-MS (BDE 209)

GC Column:

PBDEs: 30 m DB-5MS

BDE 209: 15m DB-5MS

<2 3

2 to 3 0

>3 4

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 10						Exercise	
Congener	1941b A	1941b B	1941b C	Ave.	SD	Value	z-score
BDE 15							
BDE 17							
BDE 25							
BDE 28-33	0.21	0.21	0.24	0.22	0.02	0.18	1.09
BDE 30							
BDE 33							
BDE 47	1.92	1.54	1.57	1.68	0.21	1.48	0.67
BDE 49	0.23	0.18	0.18	0.19	0.03		
BDE 66							
BDE 71							
BDE 75							
BDE 85							
BDE 99	0.62	0.56	0.59	0.59	0.03	0.62	-0.25
BDE 100	0.14	0.10	0.11	0.12	0.02	0.15	-1.09
BDE 116							
BDE 118							
BDE 119							
BDE 138							
BDE 153	0.07	0.07	0.06	0.07	0.01	0.09	-1.09
BDE 154	0.08	0.06	0.07	0.07	0.01	0.09	-1.01
BDE 155							
BDE 156							
BDE 181							
BDE 183	0.05	0.06	0.04	0.05	0.01	0.08	-1.74
BDE 190							
BDE 191							
BDE 196							
BDE 197							
BDE 203							
BDE 205							
BDE 206	3.14	1.72	1.44	2.10	0.91		
BDE 207							
BDE 208							
BDE 209	23.09	25.84	26.93	25.29	1.98	24.11	0.24

Category	z (20%)
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Instrument Used: GC/NCI-MS

GC Column

PBDEs:	15 m DB-5MS
BDE 209:	15 m DB-5MS

<2	8
2 to 3	0
>3	0

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 11				Exercise			
<u>Congener</u>	<u>1941b A</u>	<u>1941b B</u>	<u>1941b C</u>	<u>Ave.</u>	<u>SD</u>	<u>Value</u>	<u>z-score</u>
BDE 15	NA	NA	NA				
BDE 17	0.06	0.05	0.07	0.06	0.01		
BDE 25	NA	NA	NA				
BDE 28-33	DL	0.13	0.13	0.13	0.01	0.18	-1.40
BDE 30	NA	NA	NA				
BDE 33							
BDE 47	0.73	0.60	0.97	0.76	0.19	1.48	-2.42
BDE 49	NA	NA	NA				
BDE 66	DL	DL	DL				
BDE 71	DL	DL	DL				
BDE 75	NA	NA	NA				
BDE 85	DL	DL	DL				
BDE 99	DL	DL	DL			0.62	
BDE 100	DL	DL	DL			0.15	
BDE 116	NA	NA	NA				
BDE 118	NA	NA	NA				
BDE 119	NA	NA	NA				
BDE 138	DL	DL	DL				
BDE 153	DL	DL	DL			0.09	
BDE 154	DL	DL	DL			0.09	
BDE 155	NA	NA	NA				
BDE 156	NA	NA	NA				
BDE 181	NA	NA	NA				
BDE 183	DL	DL	DL			0.08	
BDE 190	DL	DL	DL				
BDE 191	NA	NA	NA				
BDE 196	NA	NA	NA				
BDE 197	NA	NA	NA				
BDE 203	NA	NA	NA				
BDE 205	NA	NA	NA				
BDE 206	NA	NA	NA				
BDE 207	NA	NA	NA				
BDE 208	NA	NA	NA				
BDE 209	DL	DL	DL			24.11	

<u>Category</u>	<u>z (20%)</u>
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Instrument Used: HRGC/NCI-MS
 GC Column
 PBDEs: 30 m DB-5
 BDE 209: 15 m DB-1

<2	1
2 to 3	1
>3	0

Table 4. Data as Submitted By Laboratory for SRM 1941b (concentration in ng/g dry mass).

Lab 12					Exercise		
Congener	1941b A	1941b B	1941b C	Ave.	SD	Value	z-score
BDE 15	NA	NA	NA				
BDE 17	0.14	0.16	0.15	0.15	0.01		
BDE 25	NA	NA	NA				
BDE 28-33	0.22	0.22	0.25	0.23	0.02	0.18	1.33
BDE 30	NA	NA	NA				
BDE 33	NA	NA	NA				
BDE 47	2.14	2.05	2.04	2.08	0.06	1.48	2.02
BDE 49	ND	ND	ND				
BDE 66	0.06	0.05	0.06	0.05	0.00		
BDE 71	ND	ND	ND				
BDE 75	NA	NA	NA				
BDE 85	ND	ND	ND				
BDE 99	0.82	0.85	0.79	0.82	0.03	0.62	1.60
BDE 100	0.23	0.22	0.22	0.22	0.00	0.15	2.36
BDE 116	NA	NA	NA				
BDE 118	NA	NA	NA				
BDE 119	ND	ND	ND				
BDE 138	ND	ND	ND				
BDE 153	0.08	0.07	0.08	0.08	0.00	0.09	-0.80
BDE 154	0.09	0.09	0.08	0.09	0.01	0.09	-0.20
BDE 155	ND	ND	ND				
BDE 156	NA	NA	NA				
BDE 181	ND	ND	ND				
BDE 183	0.05	0.04	0.05	0.05	0.00	0.08	-2.17
BDE 190	ND	ND	ND				
BDE 191	ND	ND	ND				
BDE 196	ND	ND	ND				
BDE 197	ND	ND	ND				
BDE 203	ND	ND	ND				
BDE 205	ND	ND	ND				
BDE 206	ND	ND	ND				
BDE 207	0.57	0.54	0.59	0.57	0.03		
BDE 208	ND	ND	ND				
BDE 209	146.00	144.00	146.00	145.33	1.15	24.11	25.14

Category	z (20%)
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Instrument Used: GC/NCI-MS

GC Column:

PBDEs: 60 m DB-5

BDE 209: 15 m DB-5

<2	4
2 to 3	3
>3	1

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 1						Exercise	
<u>Congener</u>	<u>1944 A</u>	<u>1944 B</u>	<u>1944 C</u>	<u>Ave.</u>	<u>SD</u>	<u>Value</u>	<u>z-score</u>
BDE 15							
BDE 17	0.62	0.659	0.664	0.65	0.02		
BDE 25							
BDE 28-33	ND	ND	ND			0.26	
BDE 30							
BDE 33							
BDE 47	2.178	2.213	2.161	2.18	0.03	1.63	1.70
BDE 49							
BDE 66	ND	ND	ND				
BDE 71	IS	IS	IS				
BDE 75							
BDE 85	0.192	0.241	0.213	0.22	0.02		
BDE 99	2.358	2.429	2.317	2.37	0.06	1.80	1.58
BDE 100	0.541	0.457	0.598	0.53	0.07	0.46	0.78
BDE 116							
BDE 118							
BDE 119							
BDE 138	0.448	0.519	0.463	0.48	0.04		
BDE 153	6.845	6.573	6.597	6.67	0.15	6.53	0.11
BDE 154	1.207	1.124	1.149	1.16	0.04	1.24	-0.32
BDE 155							
BDE 156							
BDE 181							
BDE 183	30.49	36.86	37.37	34.91	3.83	32.20	0.42
BDE 190	1.904	1.644	2.222	1.92	0.29		
BDE 191							
BDE 196							
BDE 197							
BDE 203							
BDE 205							
BDE 206							
BDE 207							
BDE 208							
BDE 209	30.251	36.856	37.37	34.83	3.97	127.53	-3.63

Instrument Used: HRGC/MS (NCI)

GC column:

PBDEs: 25 m HP-5MS
 BDE 209: 15 m HP-1

Category **z (20%)**

<2 6
 2 to 3 0
 >3 1

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 2							
Congener	1944 A	1944 B	1944 C	Ave.	SD	Exercise Value	z-score
BDE 15	NA	NA	NA				
BDE 17	1.32	1.27	1.37	1.32	0.05		
BDE 25	other	other	other				
BDE 28-33	0.18	0.2	0.22	0.20	0.02	0.26	-1.15
BDE 30	<0.1	<0.1	<0.1				
BDE 33	other	other	other				
BDE 47	1.85	1.94	1.72	1.84	0.11	1.63	0.63
BDE 49	1.79	1.68	1.75	1.74	0.06		
BDE 66	0.22	0.22	0.2	0.21	0.01		
BDE 71	<0.1	<0.1	<0.1				
BDE 75	<0.1	<0.1	<0.1				
BDE 85	0.47	0.44	0.5	0.47	0.03		
BDE 99	1.8	1.95	1.89	1.88	0.08	1.80	0.22
BDE 100	0.39	0.4	0.43	0.41	0.02	0.46	-0.58
BDE 116	<0.1	<0.1	<0.1				
BDE 118	<0.1	<0.1	<0.1				
BDE 119	<0.1	<0.1	<0.1				
BDE 138	0.77	0.72	0.85	0.78	0.07		
BDE 153	7.31	7.06	7.08	7.15	0.14	6.53	0.47
BDE 154	1.13	1.02	0.93	1.03	0.10	1.24	-0.86
BDE 155	<0.2	<0.2	<0.2				
BDE 156	<0.2	<0.2	<0.2				
BDE 181	<0.3	<0.3	<0.3				
BDE 183	26.14	32.33	27.86	28.78	3.20	32.20	-0.53
BDE 190	4.65	2.92	3.84	3.80	0.87		
BDE 191	<0.3	<0.3	<0.3				
BDE 196	33.84	21.16	27.42	27.47	6.34		
BDE 197	20.27	13.54	16.53	16.78	3.37		
BDE 203	6.43	8.53	8.93	7.96	1.34		
BDE 205	1.18	1.2	2.17	1.52	0.57		
BDE 206	16.67	10.51	14.34	13.84	3.11		
BDE 207	33.31	20.62	27.53	27.15	6.35		
BDE 208	NA	NA	NA				
BDE 209	135.6	98.3	148.7	127.53	26.15	127.53	0.00

Instrument Used: GC/MS (NCI)

GC Column:

PBDEs: 25 m HT-8
 BDE 209: 12 m AT-5

Category **z (20%)**

<2 8
 2 to 3 0
 >3 0

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 3							
<u>Congener</u>	<u>1944 A</u>	<u>1944 B</u>	<u>1944 C</u>	<u>Ave.</u>	<u>SD</u>	<u>Exercise Value</u>	<u>z-score</u>
BDE 15	NA	NA	NA				
BDE 17	NA	NA	NA				
BDE 25	NA	NA	NA				
BDE 28-33	0.12	0.13	0.13	0.13	0.01	0.26	-2.58
BDE 30	NA	NA	NA				
BDE 33	NA	NA	NA				
BDE 47	0.96	1.03	1.05	1.01	0.05	1.63	-1.89
BDE 49	NA	NA	NA				
BDE 66	NA	NA	NA				
BDE 71	NA	NA	NA				
BDE 75	NA	NA	NA				
BDE 85	NA	NA	NA				
BDE 99	1.46	1.40	1.36	1.41	0.05	1.80	-1.09
BDE 100	NA	NA	NA			0.46	
BDE 116	NA	NA	NA				
BDE 118	NA	NA	NA				
BDE 119	NA	NA	NA				
BDE 138	NA	NA	NA				
BDE 153	6.59	7.09	6.44	6.71	0.34	6.53	0.14
BDE 154	1.25	1.32	1.21	1.26	0.06	1.24	0.08
BDE 155	NA	NA	NA				
BDE 156	NA	NA	NA				
BDE 181	NA	NA	NA				
BDE 183	39.67	29.89	28.16	32.57	6.20	32.20	0.06
BDE 190	NA	NA	NA				
BDE 191	NA	NA	NA				
BDE 196	NA	NA	NA				
BDE 197	NA	NA	NA				
BDE 203	NA	NA	NA				
BDE 205	NA	NA	NA				
BDE 206	NA	NA	NA				
BDE 207	NA	NA	NA				
BDE 208	NA	NA	NA				
BDE 209	136.37	114.56	151.37	134.10	18.51	127.53	0.26

<u>Category</u>	<u>z (20%)</u>
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Instrument Used: GC/EI-MS (PBDEs); GC/ECD (BDE 209)
 GC Column: PBDEs: 30 m HP-5MS
 BDE 209: 15 m DB-5

<2	6
2 to 3	1
>3	0

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 4						Exercise	
Congener	1944 A	1944 B	1944 C	Ave.	SD	Value	z-score
BDE 15	0.95	0.98	0.97	0.97	0.01		
BDE 17	0.91	0.92	0.97	0.93	0.04		
BDE 25	other	other	other				
BDE 28-33	0.28	0.29	0.32	0.30	0.03	0.26	0.67
BDE 30	<0.020	<0.023	<0.018				
BDE 33	other	other	other				
BDE 47	1.59	1.62	1.66	1.62	0.04	1.63	-0.02
BDE 49	0.97	0.96	1.01	0.98	0.03		
BDE 66	0.20	0.20	0.28	0.23	0.05		
BDE 71	0.18	0.20	0.18	0.18	0.01		
BDE 75	0.05	0.06	0.08	0.06	0.01		
BDE 85	0.12	0.12	0.15	0.13	0.02		
BDE 99	2.10	2.03	2.36	2.16	0.17	1.80	1.01
BDE 100	0.65	0.59	0.74	0.66	0.08	0.46	2.17
BDE 116	0.12	0.11	0.13	0.12	0.01		
BDE 118	N/A	N/A	N/A				
BDE 119			<0.6				
BDE 138	0.93	0.64	0.91	0.83	0.16		
BDE 153	7.50	8.91	8.24	8.22	0.71	6.53	1.29
BDE 154	2.70	2.20	3.17	2.69	0.49	1.24	5.85
BDE 155	0.31	0.25	0.38	0.32	0.07		
BDE 156	N/A	N/A	N/A				
BDE 181	0.41	0.39	0.50	0.43	0.06		
BDE 183	30.90	44.20	35.80	36.97	6.73	32.20	0.74
BDE 190	4.30	4.26	5.49	4.68	0.70		
BDE 191	N/A	N/A	N/A				
BDE 196	N/A	N/A	N/A				
BDE 197	N/A	N/A	N/A				
BDE 203	5.09	7.17	8.70	6.99	1.81		
BDE 205	N/A	N/A	N/A				
BDE 206			6.87				
BDE 207			13.80				
BDE 208	N/A	N/A	N/A				
BDE 209	112.00	135.00	126.00	124.33	11.59	127.53	-0.13

Category **z (20%)**

Instrument Used:

GC/HRMS

GC Column:

PBDEs
BDE 209

30 m DB5 HT
30 m DB5 HT

<2 6
2 to 3 1
>3 1

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 5						Exercise	
Congener	1944 A	1944 B	1944 C	Ave.	SD	Value	z-score
BDE 15	0.62	0.64	0.64				
BDE 17	0.43	0.44	0.46	0.44	0.01		
BDE 25	NA	NA	NA				
BDE 28-33	0.15	0.16	0.16	0.16	0.01	0.26	-1.95
BDE 30	NA	NA	NA				
BDE 33	NA	NA	NA				
BDE 47	1.37	1.35	1.46	1.39	0.06	1.63	-0.73
BDE 49	1.00	1.00	0.98	0.99	0.01		
BDE 66	0.09	0.08	0.08	0.08	0.00		
BDE 71	0.13	0.13	0.12	0.13	0.01		
BDE 75	NA	NA	NA				
BDE 85	0.11	0.09	0.10	0.10	0.01		
BDE 99	1.76	1.77	1.79	1.77	0.02	1.80	-0.07
BDE 100	0.54	0.59	0.56	0.56	0.03	0.46	1.13
BDE 116	NA	NA	NA				
BDE 118	NA	NA	NA				
BDE 119	<0.008	<0.009	<0.010				
BDE 138	0.89	0.82	0.93	0.88	0.05		
BDE 153	7.20	7.16	7.24	7.20	0.04	6.53	0.51
BDE 154	0.89	0.99	0.99	0.96	0.06	1.24	-1.14
BDE 155	NA	NA	NA				
BDE 156	NA	NA	NA				
BDE 181	NA	NA	NA				
BDE 183	33.48	39.61	37.63	36.91	3.13	32.20	0.73
BDE 190	NA	NA	NA				
BDE 191	NA	NA	NA				
BDE 196	NA	NA	NA				
BDE 197	NA	NA	NA				
BDE 203	NA	NA	NA				
BDE 205	NA	NA	NA				
BDE 206	NA	NA	NA				
BDE 207	NA	NA	NA				
BDE 208	NA	NA	NA				
BDE 209	NA	NA	NA			127.53	

Category	z (20%)
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Instrument Used: GC/HRMS
 GC Column: 30 m HP-5 MS

<2	7
2 to 3	0
>3	0

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 6							
Congener	1944 A	1944 B	1944 C	Ave.	SD	Exercise Value	z-score
BDE 15							
BDE 17							
BDE 25							
BDE 28-33						0.26	
BDE 30							
BDE 33							
BDE 47	1.50	1.13	2.22	1.62	0.55	1.63	-0.04
BDE 49	0.80	0.70	0.74	0.75	0.05		
BDE 66							
BDE 71							
BDE 75							
BDE 85							
BDE 99						1.80	
BDE 100						0.46	
BDE 116							
BDE 118							
BDE 119							
BDE 138							
BDE 153	6.09	4.87	6.06	5.67	0.70	6.53	-0.66
BDE 154						1.24	
BDE 155							
BDE 156							
BDE 181							
BDE 183	22.50	21.60	21.20	21.77	0.67	32.20	-1.62
BDE 190							
BDE 191							
BDE 196							
BDE 197							
BDE 203	25.20	26.00	27.20	26.13	1.01		
BDE 205							
BDE 206	5.49	5.72	5.88	5.70	0.20		
BDE 207							
BDE 208							
BDE 209	87.60	103.00	83.90	91.50	10.13	127.53	-1.41

Category	z (20%)
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Instrument Used: GC/MS -EI: GC/NCI-MS (BDE 209)

GC Column:

PBDEs: 30 m DB-5MS

BDE 209: 15 m DB-5MS

<2	4
2 to 3	0
>3	0

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 7						Exercise	
<u>Congener</u>	<u>1944 A</u>	<u>1944 B</u>	<u>1944 C</u>	<u>Ave.</u>	<u>SD</u>	<u>Value</u>	<u>z-score</u>
BDE 15	NA	NA	NA				
BDE 17	other	other	other				
BDE 25	other	other	other				
BDE 28-33	other	other	other			0.26	
BDE 30	other	other	other				
BDE 33	other	other	other				
BDE 47	other	other	other			1.63	
BDE 49	other	other	other				
BDE 66	other	other	other				
BDE 71	other	other	other				
BDE 75	other	other	other				
BDE 85	other	other	other				
BDE 99	other	other	other			1.80	
BDE 100	other	other	other			0.46	
BDE 116	other	other	other				
BDE 118	NA	NA	NA				
BDE 119	other	other	other				
BDE 138	other	other	other				
BDE 153	other	other	other			6.53	
BDE 154	other	other	other			1.24	
BDE 155	other	other	other				
BDE 156	other	other	other				
BDE 181	other	other	other				
BDE 183	other	other	other			32.20	
BDE 190	other	other	other				
BDE 191	other	other	other				
BDE 196	NA	NA	NA				
BDE 197	NA	NA	NA				
BDE 203	other	other	other				
BDE 205	other	other	other				
BDE 206	4.12	2.01	1.41	2.51	1.43		
BDE 207	55.30	27.60	30.00	37.63	15.35		
BDE 208	3.71	1.85	1.59	2.38	1.16		
BDE 209	90.40	255.00	82.10	142.50	97.52	127.53	0.59

<u>Category</u>	<u>z (20%)</u>
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Instrument Used: GC/NCI-MS
 GC Column

PBDEs: 20 m DB-1MS
 BDE 209: 20 m DB-1MS

<2	1
2 to 3	0
>3	0

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 8						Exercise	
Congener	1944 A	1944 B	1944 C	Ave.	SD	Value	z-score
BDE 15	NA	NA					
BDE 17	NA	NA					
BDE 25	NA	NA					
BDE 28-33	<0,1	<0,1				0.26	
BDE 30	NA	NA					
BDE 33	NA	NA					
BDE 47	1.59	1.65		1.62	0.04	1.63	-0.02
BDE 49	1.39	1.25		1.32	0.09		
BDE 66	< 0,1	< 0,1					
BDE 71	0.17	0.16		0.17	0.00		
BDE 75	<0,1	<0,1					
BDE 85	0.38	0.30		0.34	0.06		
BDE 99	1.31	1.48		1.39	0.12	1.80	-1.13
BDE 100	0.30	0.34		0.32	0.03	0.46	-1.53
BDE 116	<0,1	<0,1					
BDE 118	NA	NA					
BDE 119	NA	NA					
BDE 138	<0,1	<0,1					
BDE 153	8.60	8.92		8.76	0.23	6.53	1.71
BDE 154	1.01	1.01		1.01	0.00	1.24	-0.91
BDE 155	NA	NA					
BDE 156	NA	NA					
BDE 181	IS	IS					
BDE 183	44.54	52.64		48.59	5.73	32.20	2.54
BDE 190	<1	<1					
BDE 191	NA	NA					
BDE 196	Other	Other					
BDE 197	Other	Other					
BDE 203	Other	Other					
BDE 205	Other	Other					
BDE 206	3.52	5.14		4.33	1.14		
BDE 207	35.82	25.61		30.72	7.22		
BDE 208	NA	NA					
BDE 209	104.40	158.83		131.61	38.49	127.53	0.16

Category	z (20%)
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Instrument Used: GC/NCI-MS

GC Column:

PBDEs:	30 m RTx_CLPesticides
BDE 209:	30 m RTx_CLPesticides

<2	6
2 to 3	1
>3	0

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 9							
Congener	1944 A	1944 B	1944 C	Ave.	SD	Exercise Value	z-score
BDE 15	NA	NA	NA				
BDE 17	NA	NA	NA				
BDE 25	NA	NA	NA				
BDE 28-33	0.78	0.73	0.86	0.79	0.07	0.26	10.22
BDE 30	NA	NA	NA				
BDE 33	NA	NA	NA				
BDE 47	2.14	1.76	2.66	2.19	0.45	1.63	1.71
BDE 49	NA	NA	NA				
BDE 66	DL	DL	DL				
BDE 71	NA	NA	NA				
BDE 75	NA	NA	NA				
BDE 85	DL	DL	DL				
BDE 99	1.82	1.70	1.40	1.64	0.22	1.80	-0.44
BDE 100	0.59	0.43	0.53	0.51	0.08	0.46	0.59
BDE 116	NA	NA	NA				
BDE 118	NA	NA	NA				
BDE 119	NA	NA	NA				
BDE 138	NA	NA	NA				
BDE 153	6.38	5.53	5.65	5.85	0.46	6.53	-0.52
BDE 154	0.98	1.46	1.13	1.19	0.25	1.24	-0.21
BDE 155	NA	NA	NA				
BDE 156	NA	NA	NA				
BDE 181	NA	NA	NA				
BDE 183	32.60	30.90	30.70	31.40	1.04	32.20	-0.12
BDE 190	NA	NA	NA				
BDE 191	NA	NA	NA				
BDE 196	NA	NA	NA				
BDE 197	NA	NA	NA				
BDE 203	NA	NA	NA				
BDE 205	NA	NA	NA				
BDE 206	NA	NA	NA				
BDE 207	NA	NA	NA				
BDE 208	NA	NA	NA				
BDE 209	389.00	299.00	338.00	342.00	45.13	127.53	8.41

Category	z (20%)
-----------------	----------------

Instrument Used: GC/EI-MS (PBDEs); GC/NCI-MS (BDE 209)

GC Column:

PBDEs: 30 m DB-5MS

BDE 209: 15m DB-5MS

<2	6
2 to 3	0
>3	2

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 10						Exercise	
Congener	1944 A	1944 B	1944 C	Ave.	SD	Value	z-score
BDE 15							
BDE 17							
BDE 25							
BDE 28-33						0.26	
BDE 30							
BDE 33							
BDE 47	2.22	2.69	2.00	2.30	0.35	1.63	2.06
BDE 49	1.54	2.71	2.39	2.21	0.60		
BDE 66							
BDE 71							
BDE 75							
BDE 85							
BDE 99	2.12	2.76	1.80	2.22	0.49	1.80	1.18
BDE 100	0.43	0.70	0.38	0.50	0.18	0.46	0.47
BDE 116							
BDE 118							
BDE 119							
BDE 138							
BDE 153	5.45	6.25	4.84	5.52	0.71	6.53	-0.78
BDE 154	2.05	2.02	1.93	2.00	0.06	1.24	3.06
BDE 155							
BDE 156							
BDE 181							
BDE 183	36.16	31.89	21.33	29.79	7.63	32.20	-0.37
BDE 190							
BDE 191							
BDE 196							
BDE 197							
BDE 203	14.16	9.02	5.43	9.54	4.39		
BDE 205							
BDE 206	12.81	11.77	8.46	11.02	2.27		
BDE 207							
BDE 208							
BDE 209	121.71	137.55	99.84	119.70	18.93	127.53	-0.31

Category	z (20%)
-----------------	----------------

Instrument Used: GC/NCI-MS

<2 5

GC Column

2 to 3 2

PBDEs: 15 m DB-5MS

>3 0

BDE 209: 15 m DB-5MS

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 11							
<u>Congener</u>	<u>1944 A</u>	<u>1944 B</u>	<u>1944 C</u>	<u>Ave.</u>	<u>SD</u>	<u>Exercise Value</u>	<u>z-score</u>
BDE 15	NA	NA	NA				
BDE 17	0.47	0.39	0.35	0.40	0.06		
BDE 25	NA	NA	NA				
BDE 28-33	0.55	0.51	0.46	0.51	0.05	0.26	4.71
BDE 30	NA	NA	NA				
BDE 33							
BDE 47	1.35	1.46	1.30	1.37	0.08	1.63	-0.80
BDE 49	NA	NA	NA				
BDE 66	DL	DL	DL				
BDE 71	0.65	0.97	0.15	0.59	0.41		
BDE 75	NA	NA	NA				
BDE 85	DL	DL	DL				
BDE 99	1.48	1.54	1.44	1.49	0.05	1.80	-0.87
BDE 100	0.31	0.35	0.24	0.30	0.06	0.46	-1.74
BDE 116	NA	NA	NA				
BDE 118	NA	NA	NA				
BDE 119	NA	NA	NA				
BDE 138	0.43	0.43	0.54	0.47	0.06		
BDE 153	3.77	4.58	3.97	4.11	0.42	6.53	-1.86
BDE 154	0.67	0.87	0.58	0.71	0.15	1.24	-2.15
BDE 155	NA	NA	NA				
BDE 156	NA	NA	NA				
BDE 181	NA	NA	NA				
BDE 183	21.70	23.40	19.10	21.40	2.17	32.20	-1.68
BDE 190	1.66	1.65	0.99	1.43	0.38		
BDE 191	NA	NA	NA				
BDE 196	NA	NA	NA				
BDE 197	NA	NA	NA				
BDE 203	NA	NA	NA				
BDE 205	NA	NA	NA				
BDE 206	NA	NA	NA				
BDE 207	NA	NA	NA				
BDE 208	NA	NA	NA				
BDE 209	41.20	45.30	46.70	44.40	2.86	127.53	-3.26

<u>Category</u>	<u>z (20%)</u>
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Instrument Used: HRGC/NCI-MS

GC Column:

PBDEs:	30 m DB-5
BDE 209:	15 m DB-1

<2	5
2 to 3	1
>3	2

Table 5. Data as Submitted By Laboratory for SRM 1944 (concentration in ng/g dry mass).

Lab 12						Exercise	
Congener	1944 A	1944 B	1944 C	Ave.	SD	Value	z-score
BDE 15	NA	NA	NA				
BDE 17	0.39	0.36	0.41	0.39	0.02		
BDE 25	NA	NA	NA				
BDE 28-33	0.18	0.17	0.17	0.17	0.00	0.26	-1.66
BDE 30	NA	NA	NA				
BDE 33	NA	NA	NA				
BDE 47	1.31	1.36	1.30	1.32	0.03	1.63	-0.94
BDE 49							
BDE 66	0.06	0.07	0.08	0.07	0.01		
BDE 71							
BDE 75	NA	NA	NA				
BDE 85	0.12	0.12	0.11	0.12	0.01		
BDE 99	1.91	1.91	1.96	1.92	0.03	1.80	0.35
BDE 100	0.46	0.48	0.47	0.47	0.01	0.46	0.13
BDE 116	NA	NA	NA				
BDE 118	NA	NA	NA				
BDE 119	ND	ND	ND				
BDE 138	0.43	0.49	0.47	0.46	0.03		
BDE 153	7.26	7.71	7.16	7.38	0.29	6.53	0.65
BDE 154	1.32	1.41	1.31	1.35	0.06	1.24	0.44
BDE 155	ND	ND	ND				
BDE 156	NA	NA	NA				
BDE 181	ND	ND	ND				
BDE 183	43.33	40.16	39.02	40.84	2.23	32.20	1.34
BDE 190	3.52	3.27	3.13	3.31	0.20		
BDE 191	ND	ND	ND				
BDE 196	19.10	18.40	20.70	19.40	1.18		
BDE 197	9.08	10.10	10.20	9.79	0.62		
BDE 203	7.08	6.08	6.87	6.68	0.53		
BDE 205	ND	ND	ND				
BDE 206	9.15	9.86	8.80	9.27	0.54		
BDE 207	36.60	32.70	39.30	36.20	3.32		
BDE 208	2.10	2.07	1.72	1.96	0.21		
BDE 209	1514.00	1400.00	1486.00	1466.67	59.41	127.53	52.50

Category	z (20%)
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Instrument Used:	GC/NCI-MS
GC Column:	
PBDEs:	60 m DB-5
BDE 209:	15 m DB-5

<2	7
2 to 3	0
>3	1

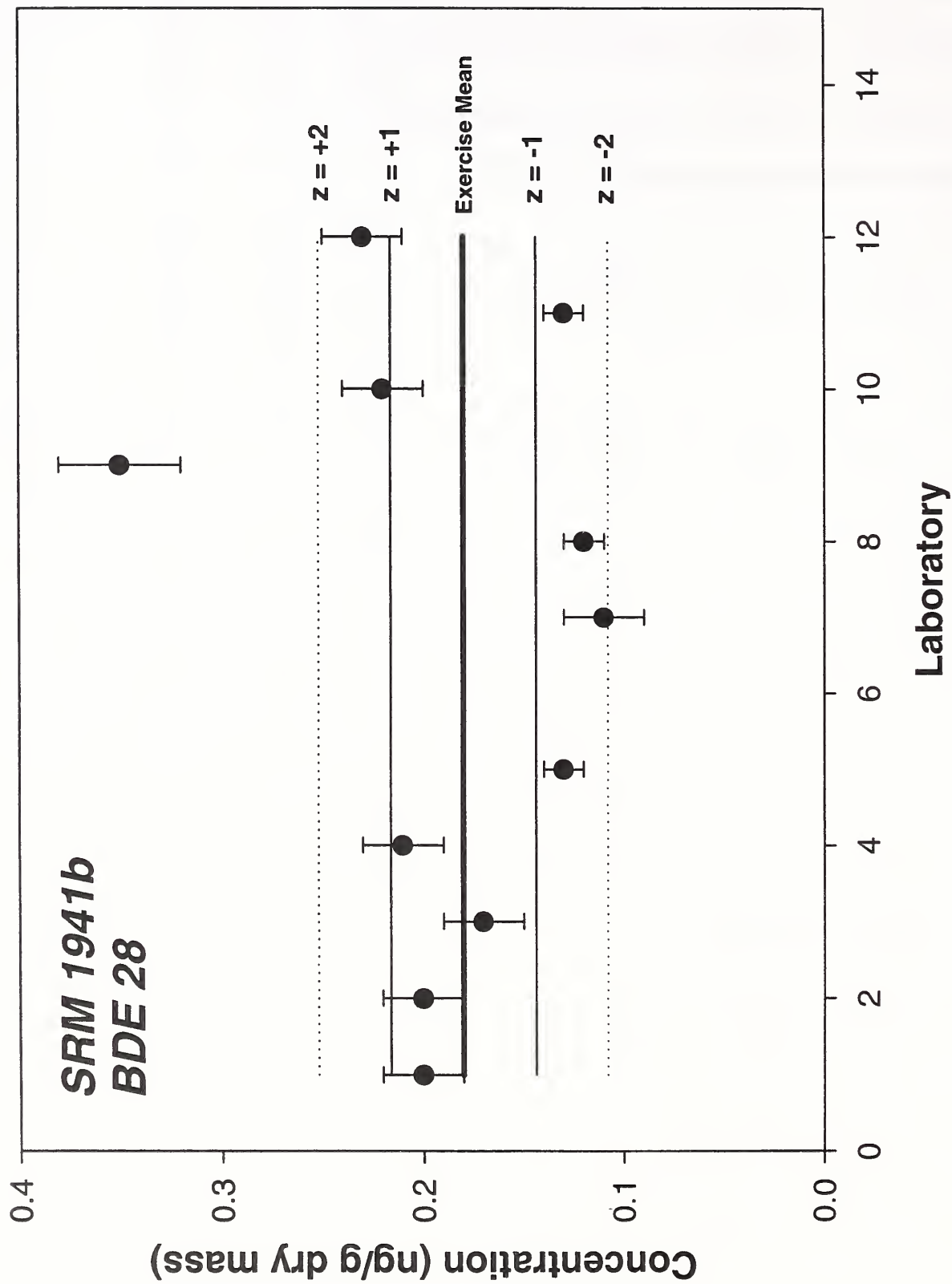
Appendix A: Charts of SRM 1941b and SRM 1944 by BDE congener

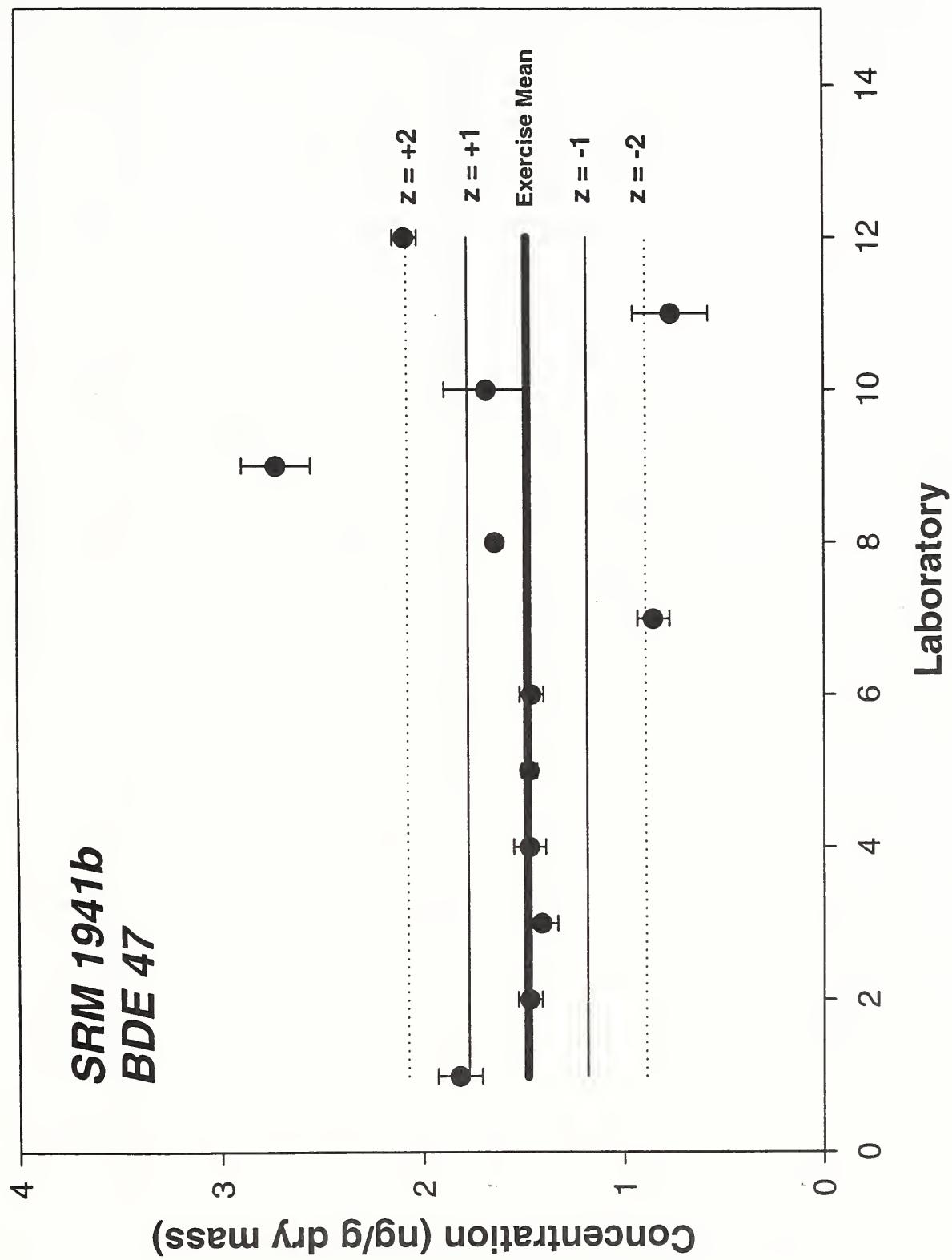
Solid line: exercise assigned value (geometric mean)

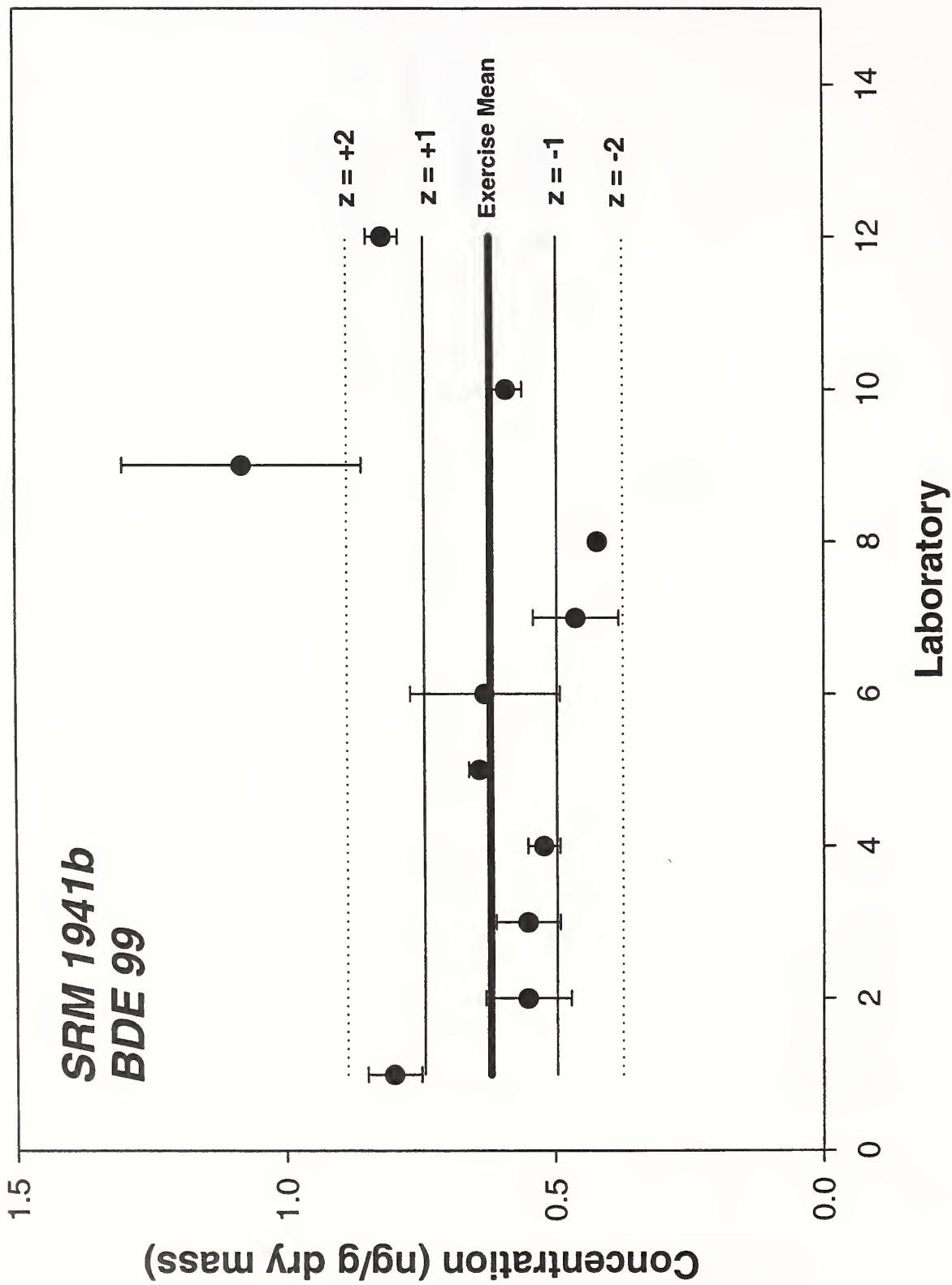
Thin line: $z = \pm 1$, i.e., 20% from assigned value

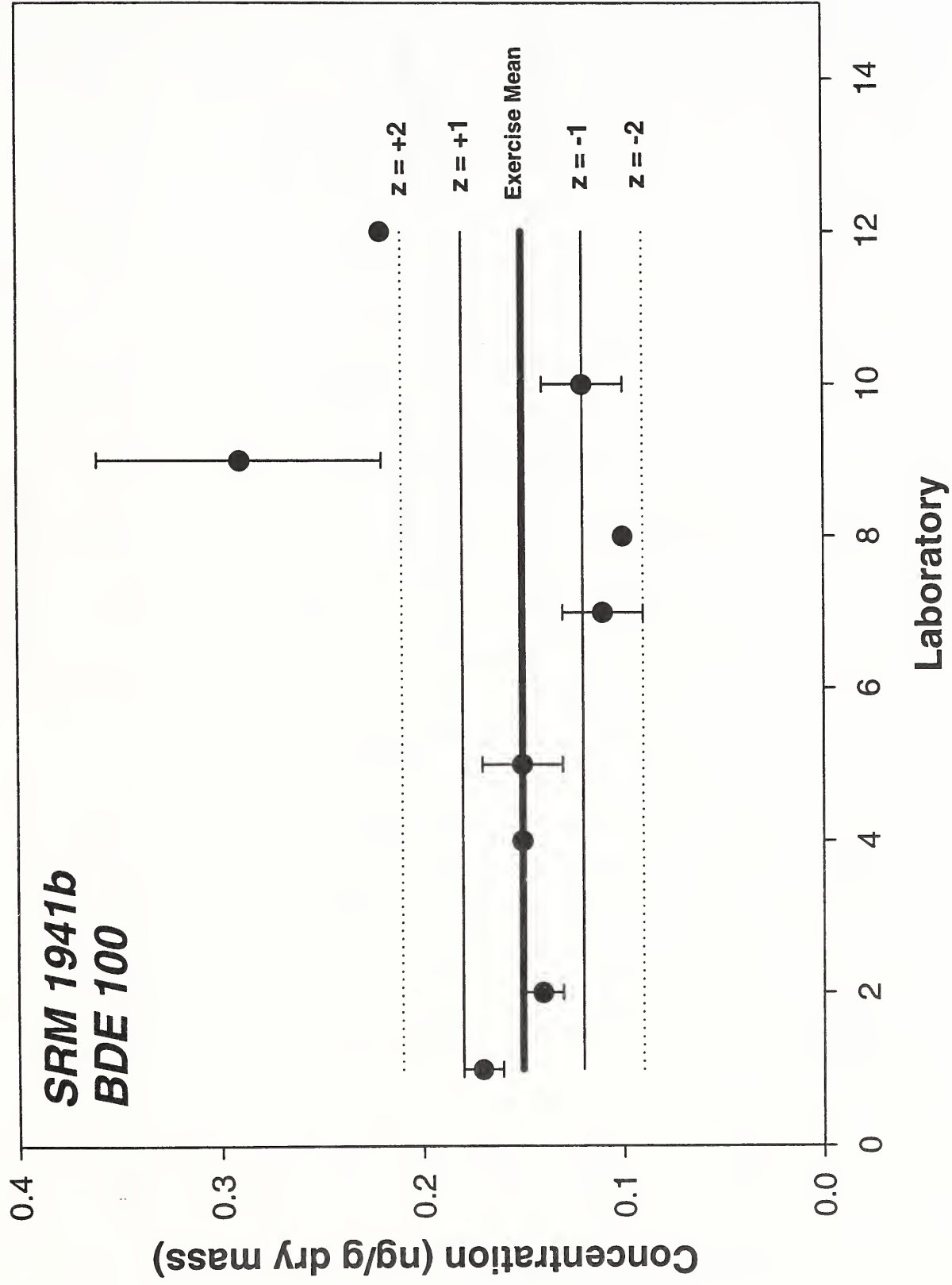
Dashed line $z = \pm 2$ i.e., 40% from assigned value

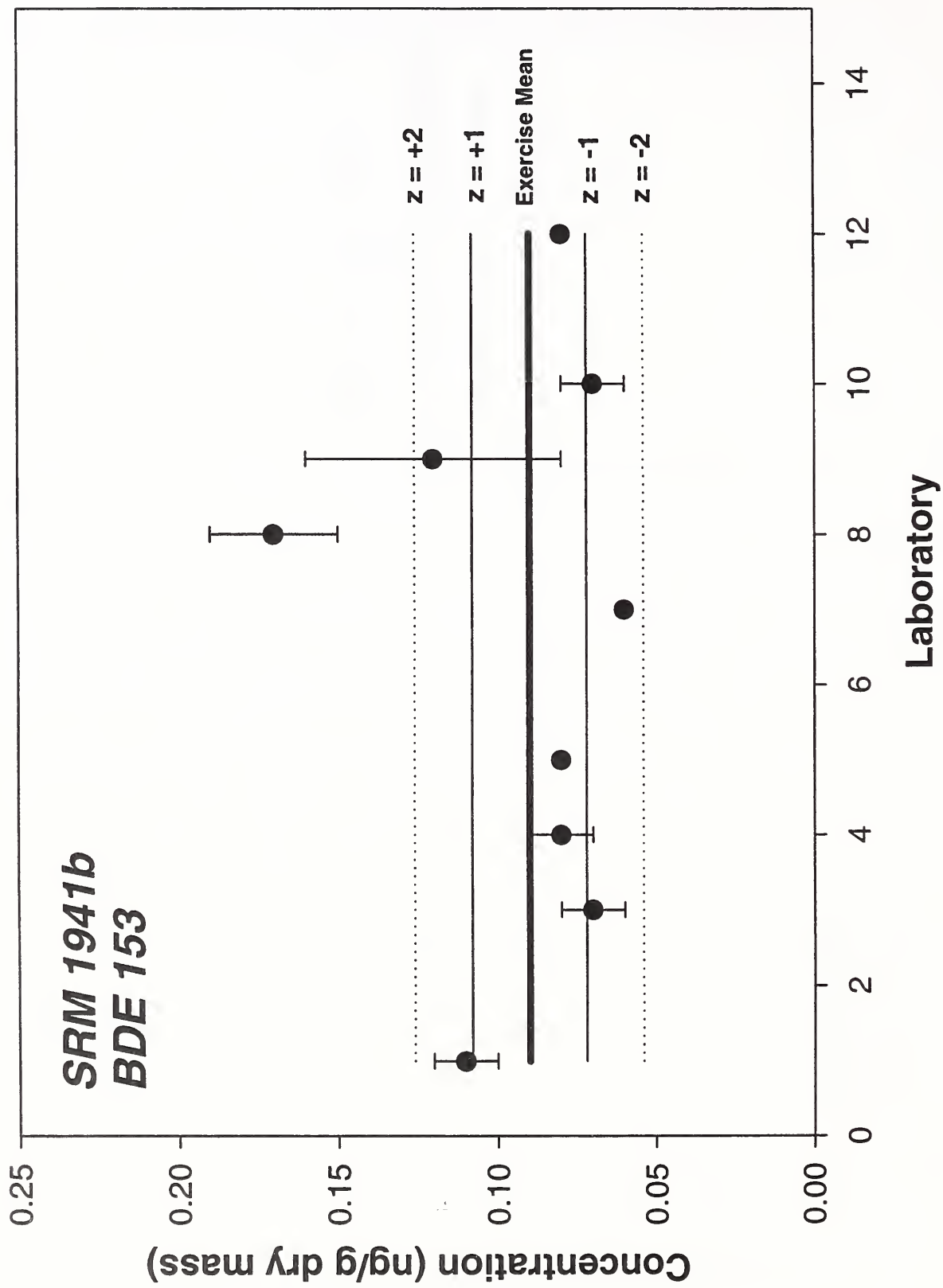
Error bars represent one standard deviation

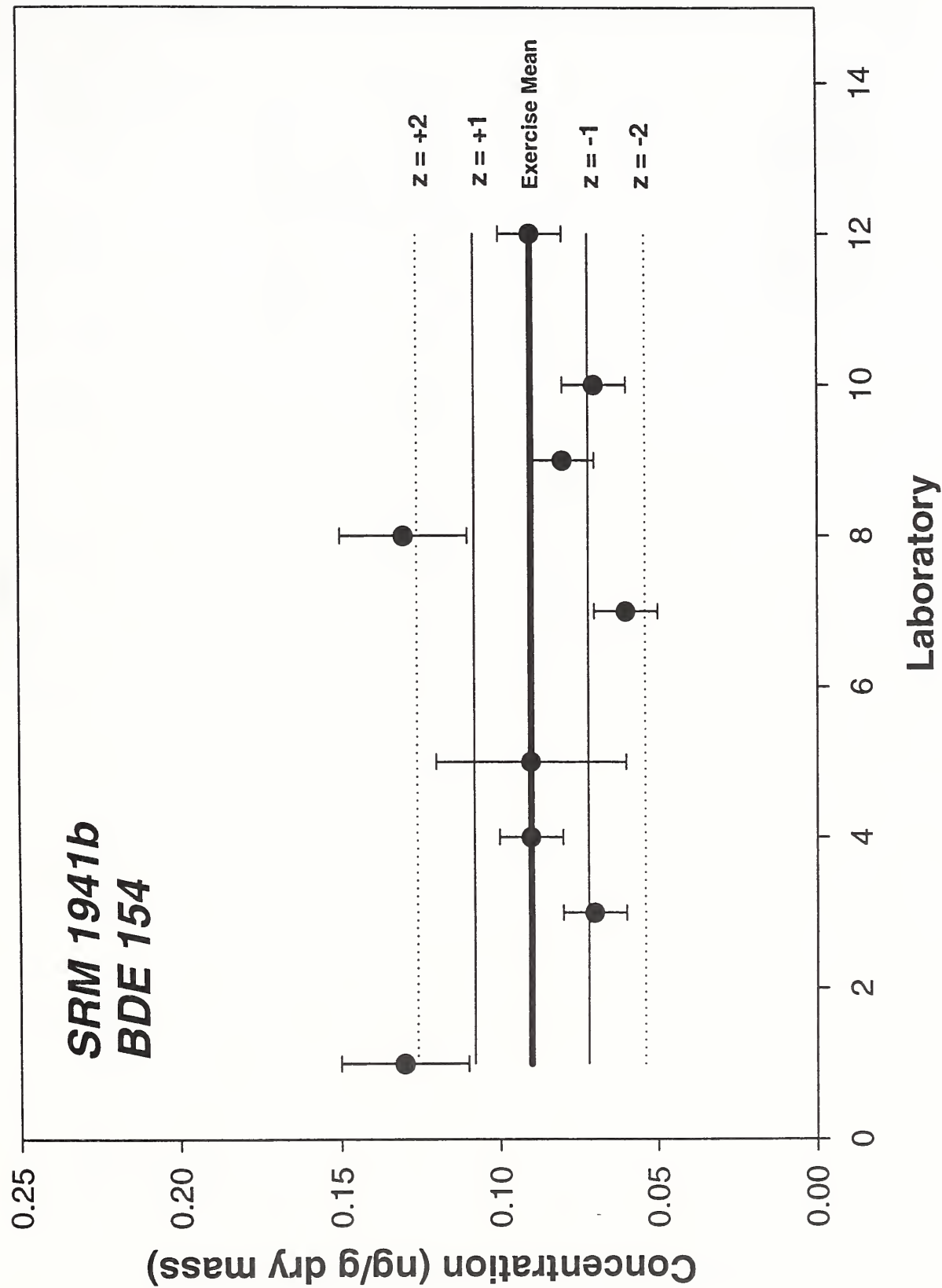


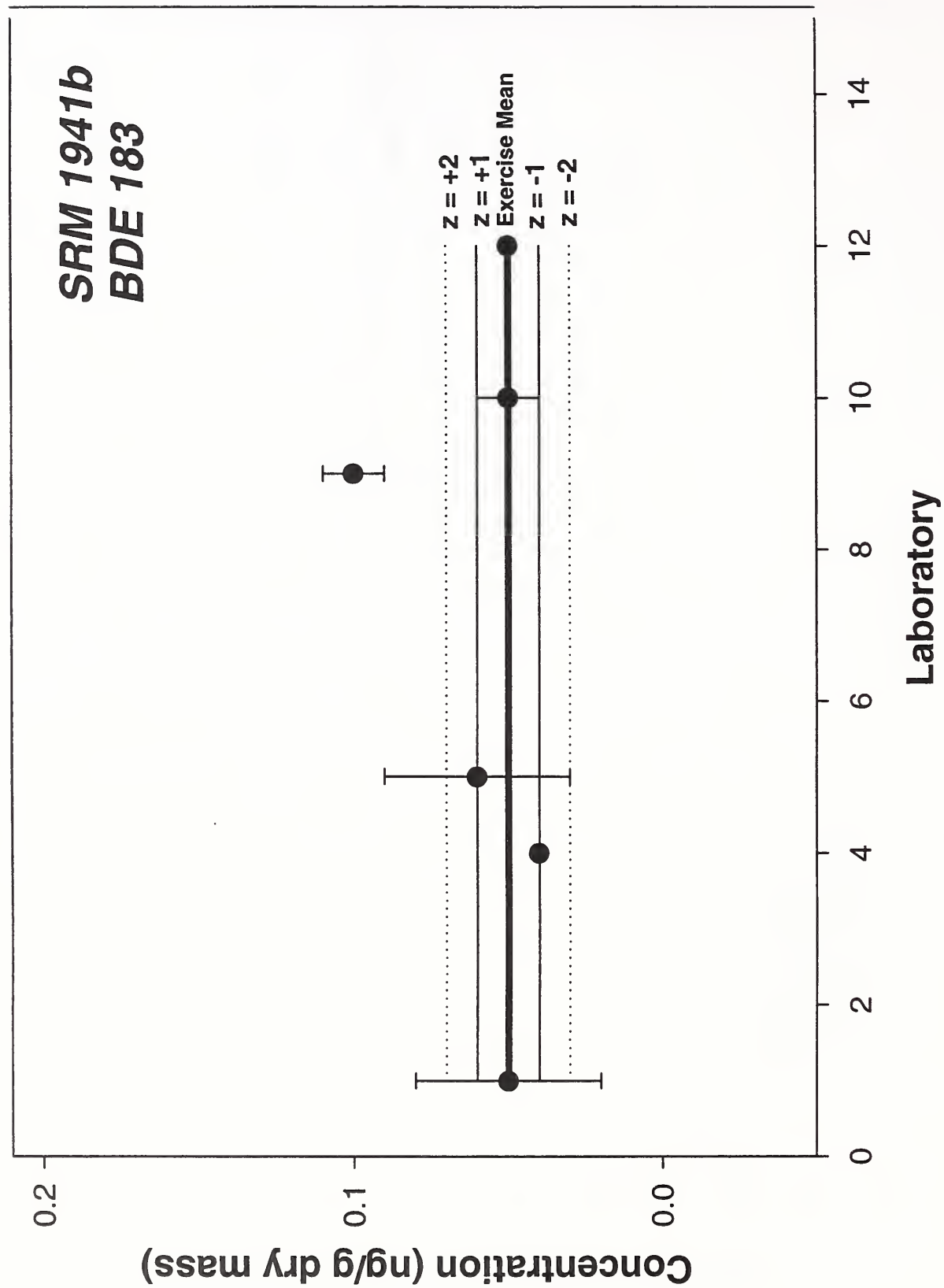






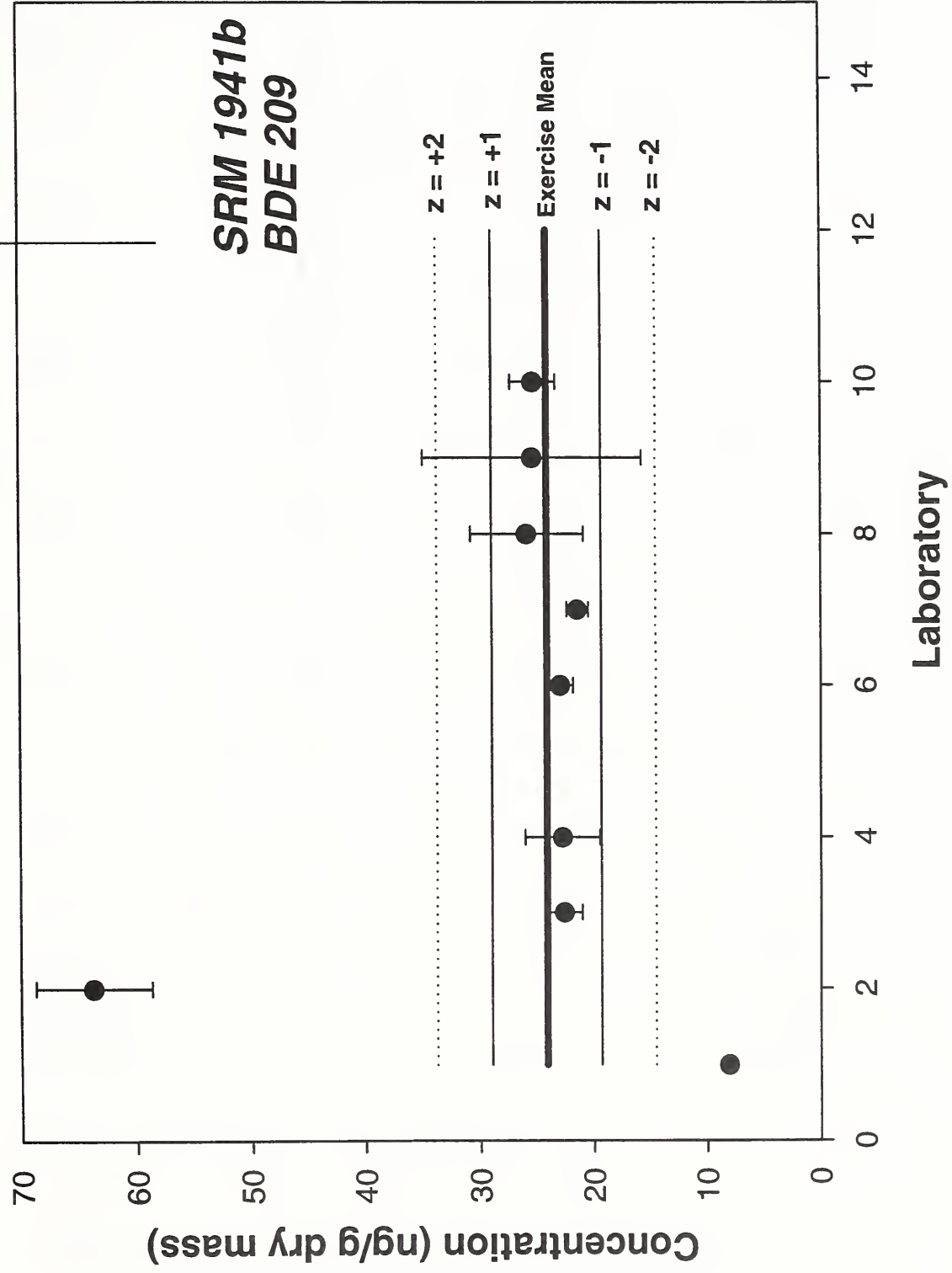


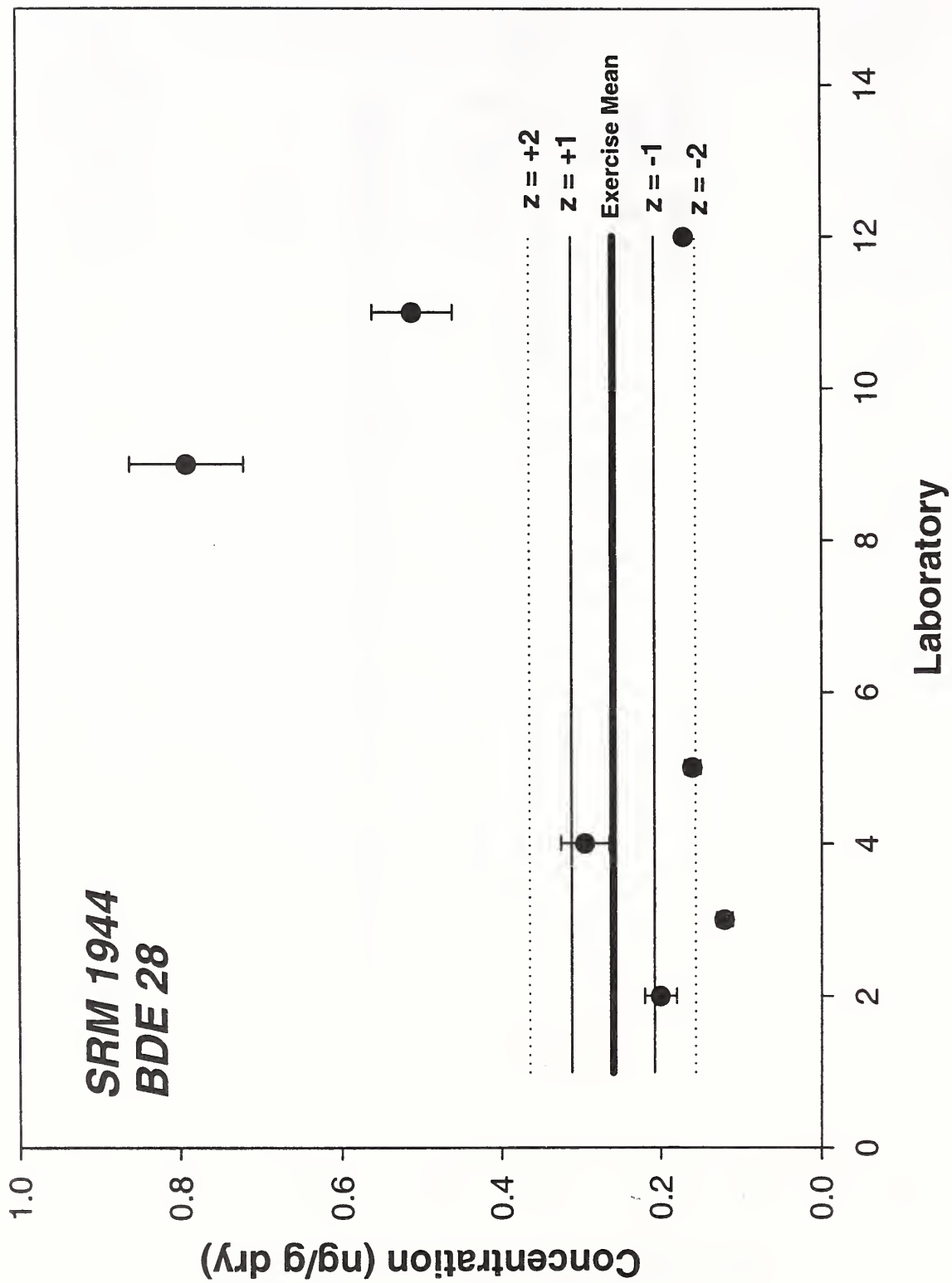


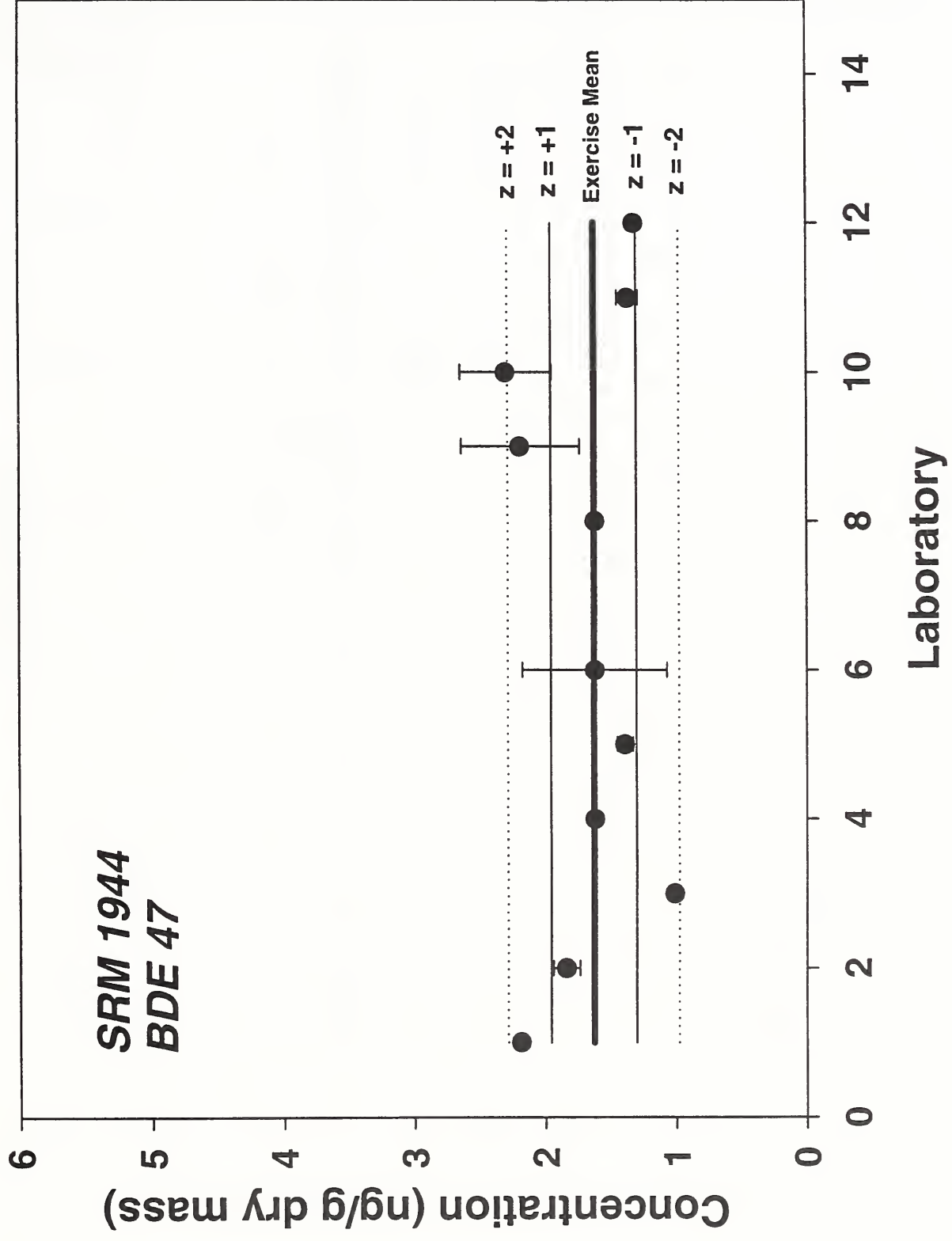


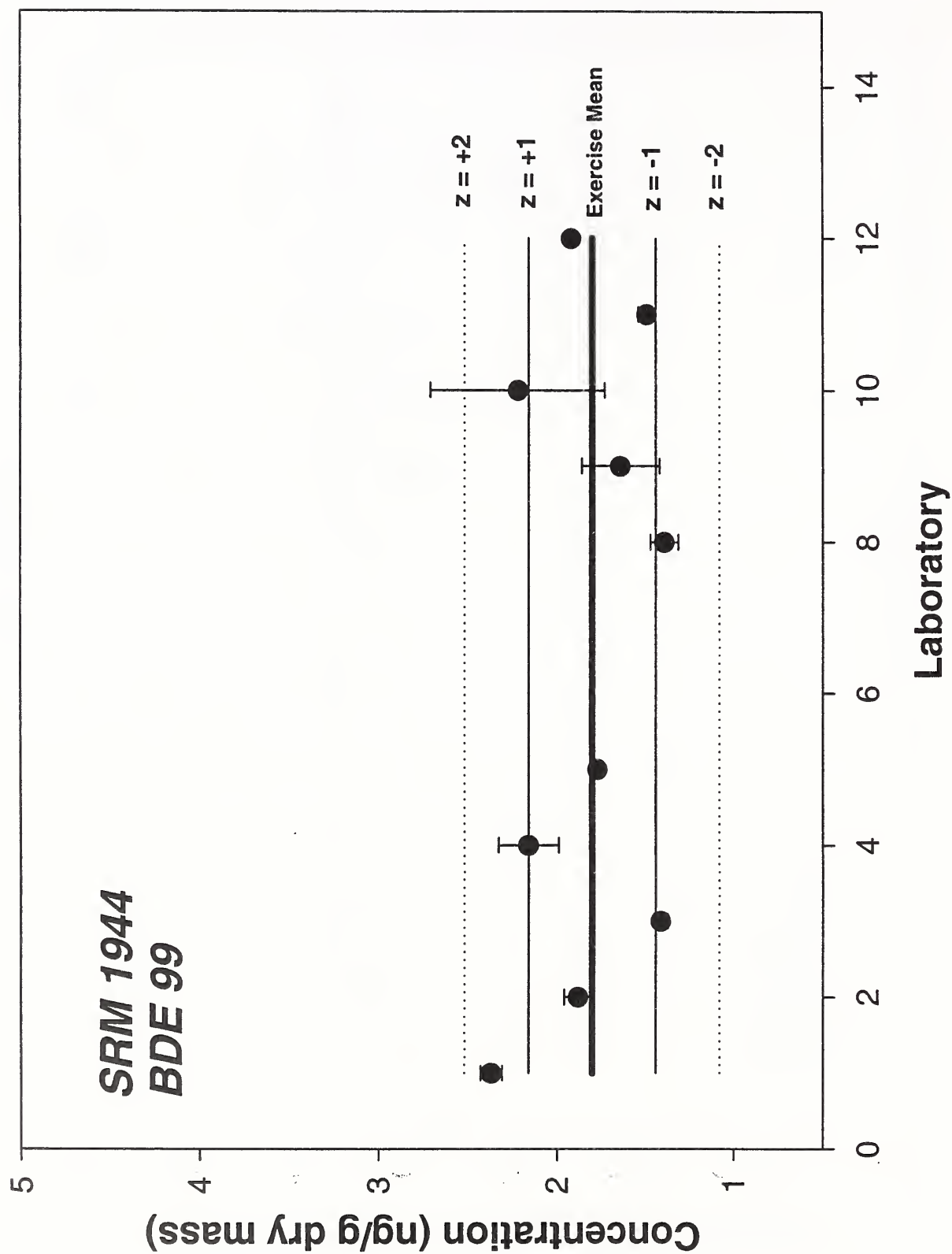
Lab # 12
145.33

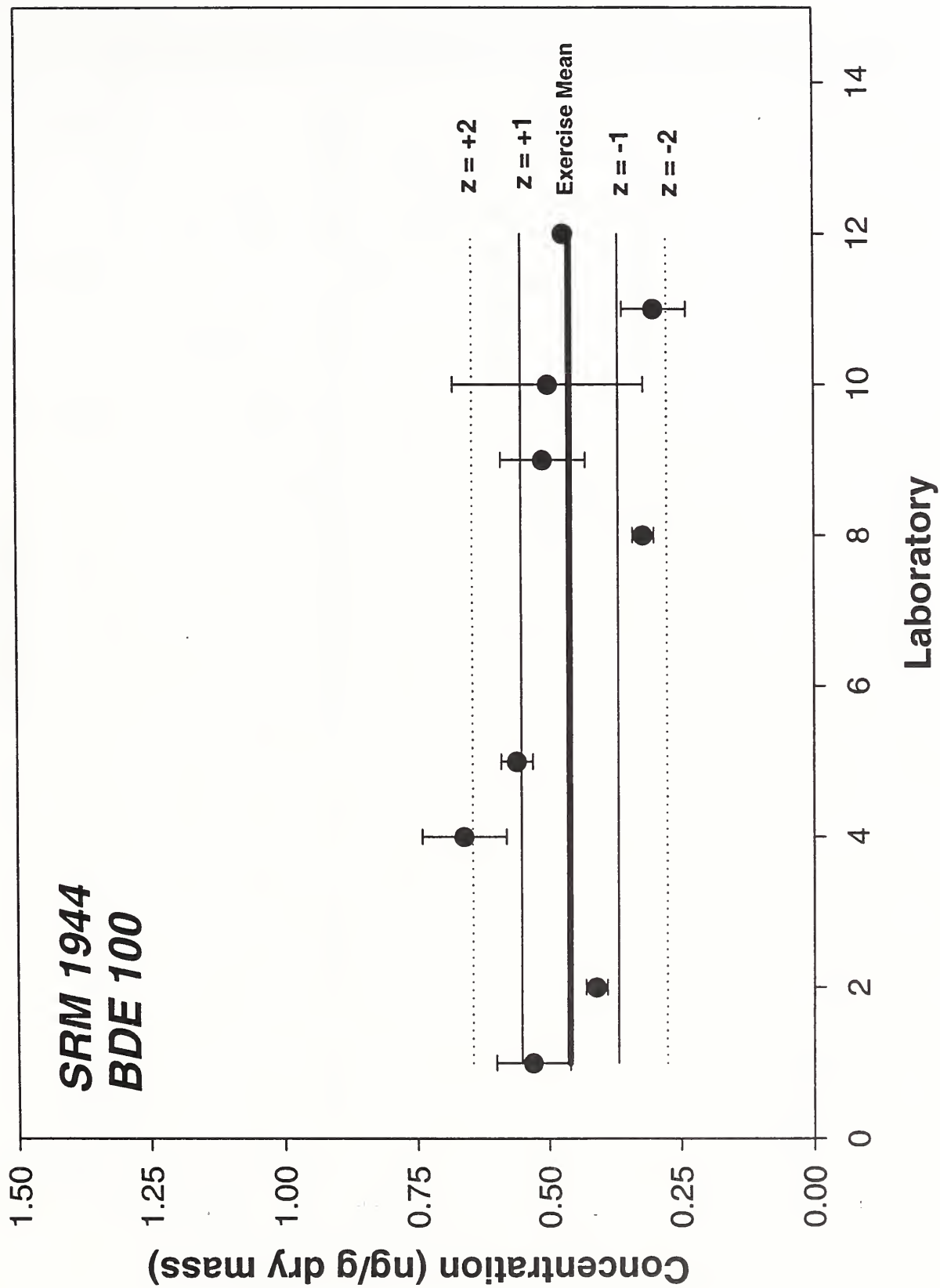
SRM 1941b
BDE 209

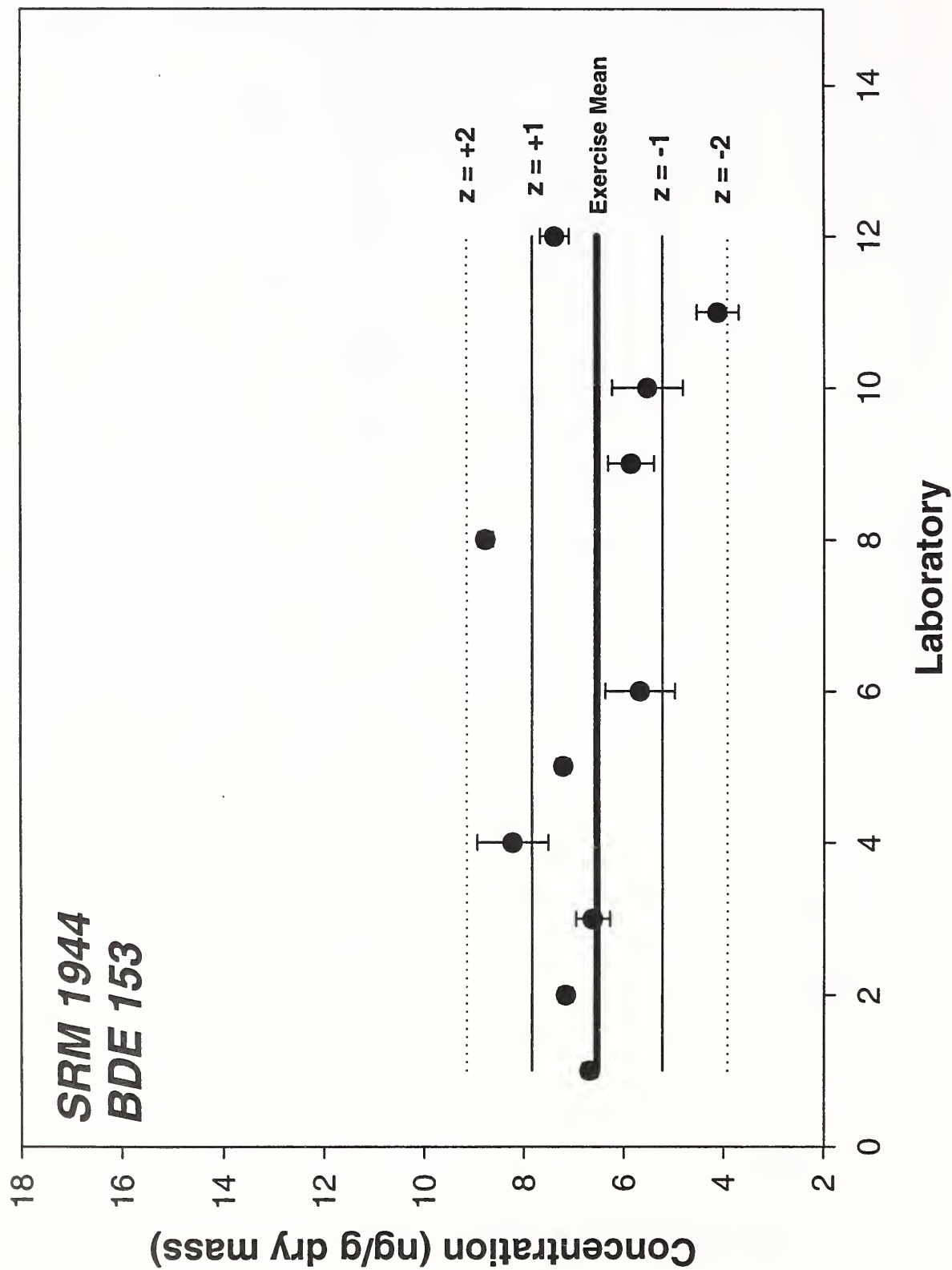


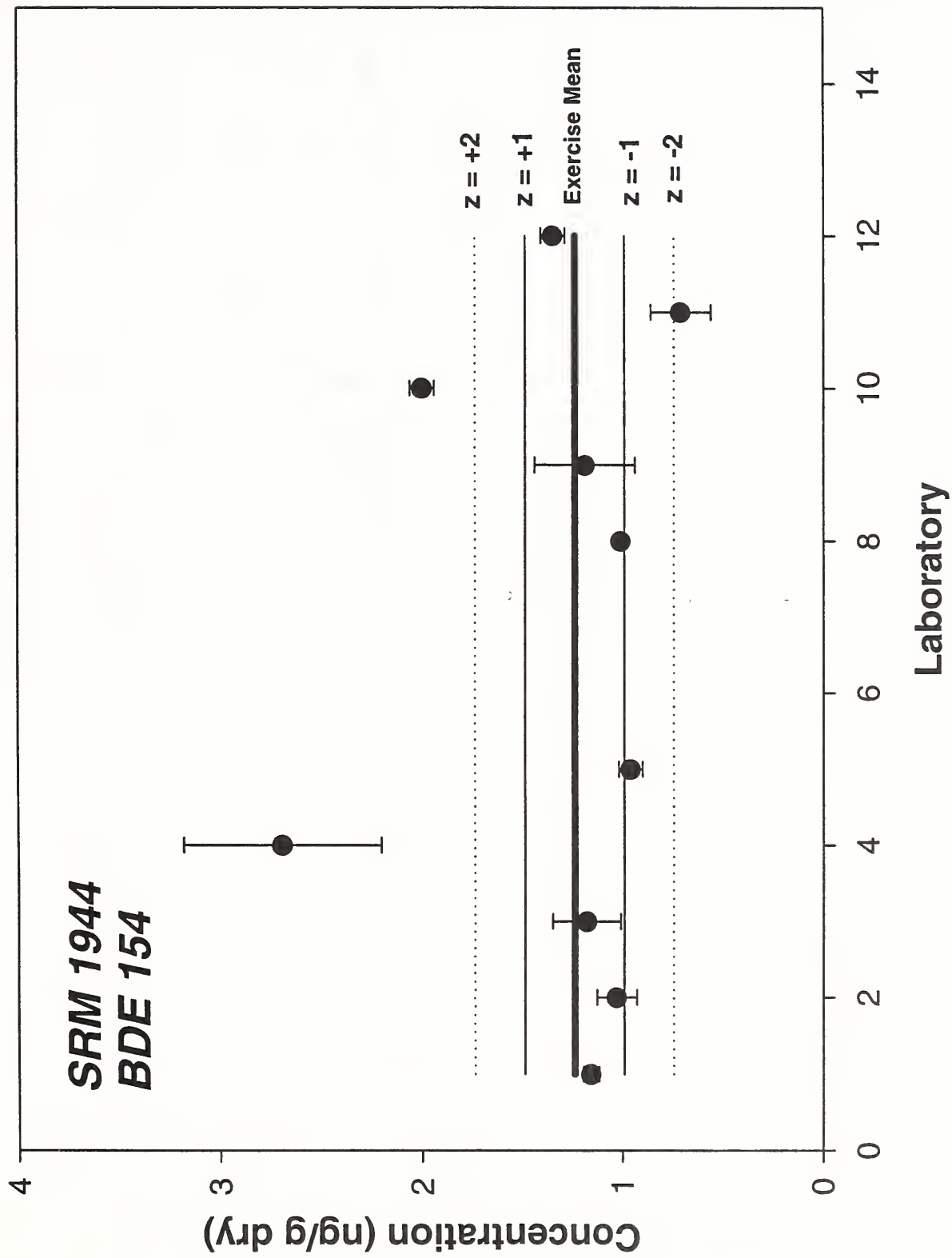


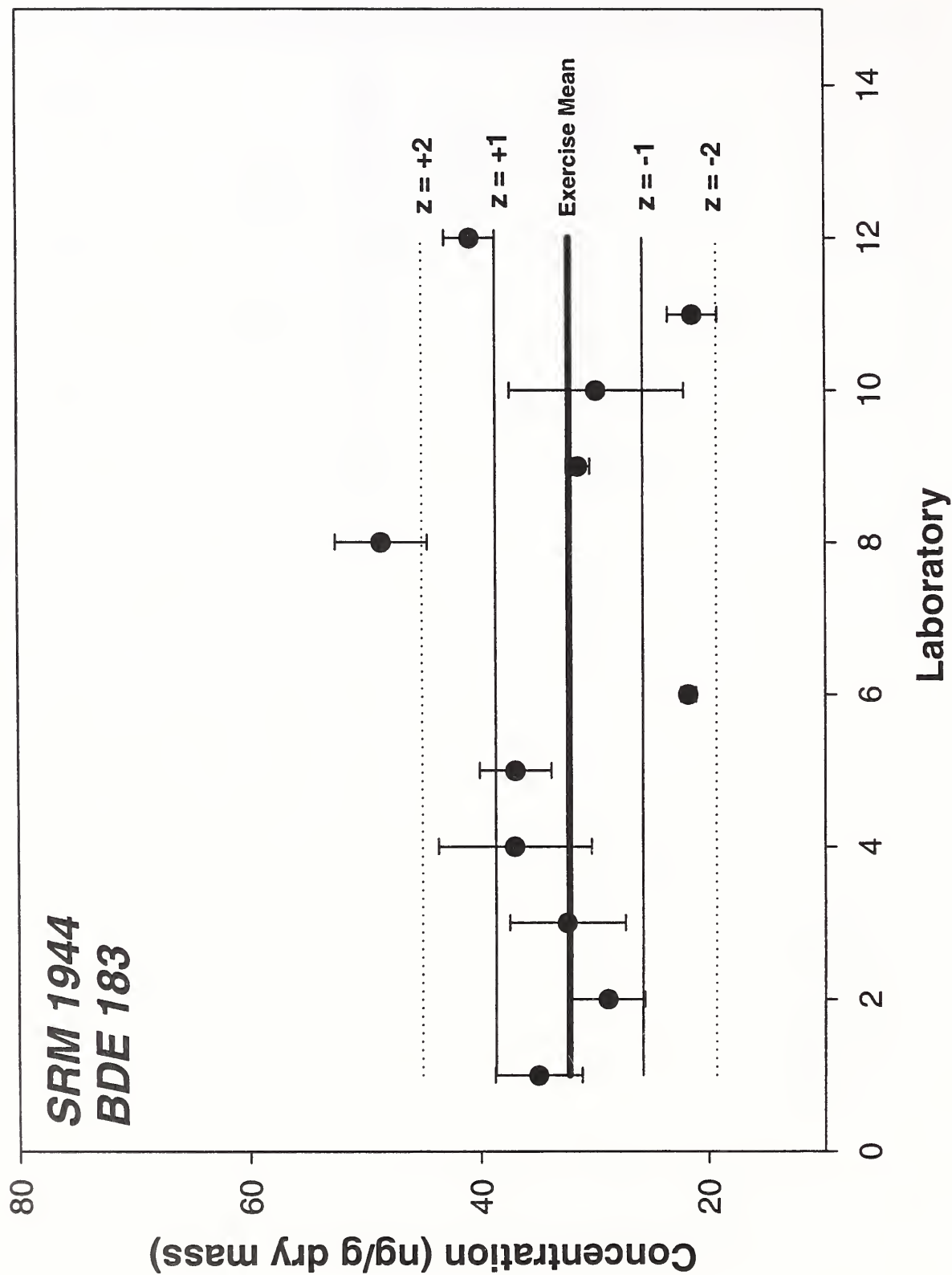


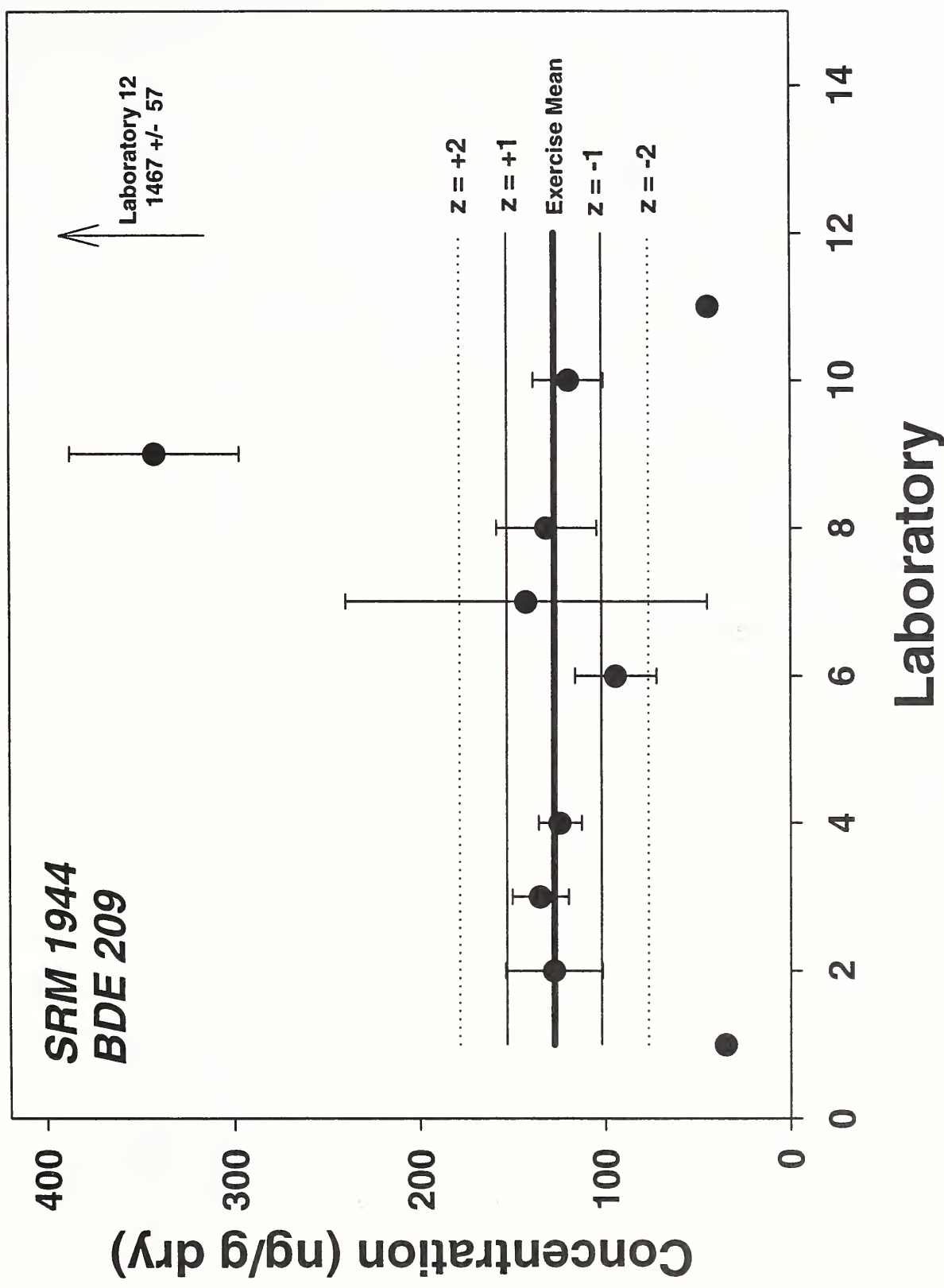






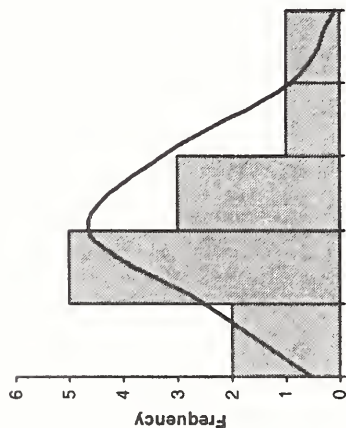




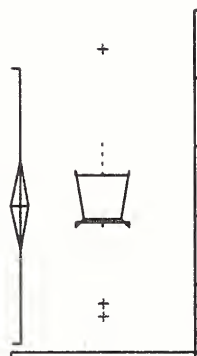


Appendix B: Results from statistical analyses of data received. Results are reported on a congener basis for each SRM.

SRM 1941b
BDE 47 Results



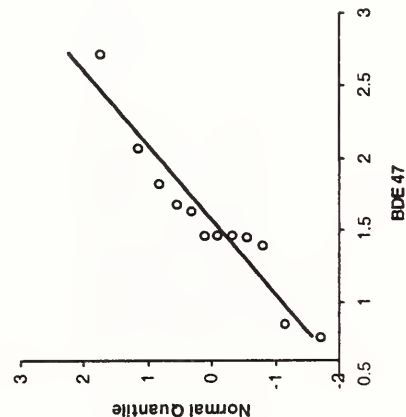
n	12
Mean	1.569
95% CI	1.243 to 1.895
Variance	0.2627
SD	0.5126
SE	0.1480
CV	33%



Median	1.471
96.1% CI	1.406 to 1.823

Range	1.96
IQR	0.34

Percentile	
2.5th	-
25th	1.449
50th	1.471
75th	1.786
97.5th	-



	Coefficient	p
Shapiro-Wilk	0.9135	0.2365
Skewness	0.6300	0.3077
Kurtosis	1.6931	-

SRM 1941b
BDE 99 Results

n 11 (cases excluded: 1 due to missing values)

Mean 0.643
95% CI 0.514 to 0.771

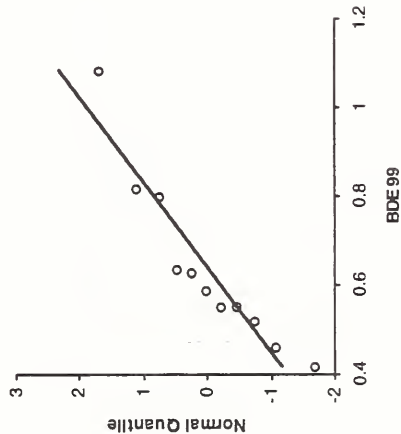
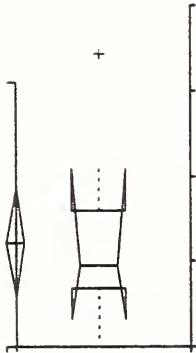
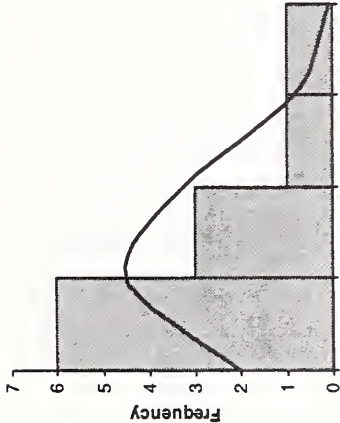
Variance 0.0366
SD 0.1914
SE 0.0577
CV 30%

Median 0.589
98.8% CI 0.463 to 0.818

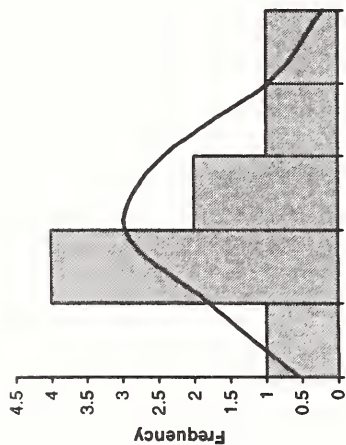
Range 0.67
IQR 0.18

Percentile	
2.5th	-
25th	0.537
50th	0.589
75th	0.719
97.5th	-

	Coefficient	P
Shapiro-Wilk	0.8877	0.1302
Skewness	1.3148	0.0490
Kurtosis	1.7892	-



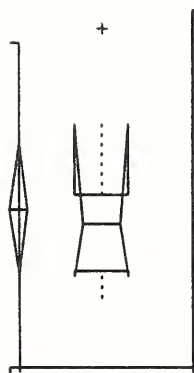
SRm 1941b
BDE 100 Results



n 9 (cases excluded: 3 due to missing values)

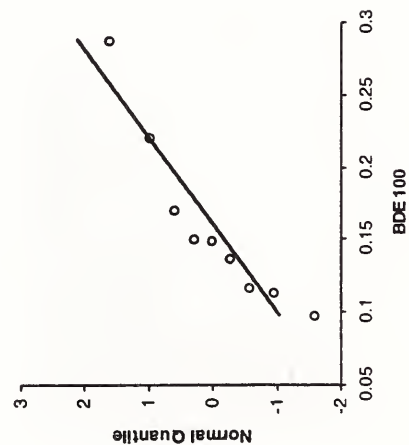
Mean 0.160
95% CI 0.115 to 0.206

Variance 0.0036
SD 0.0598
SE 0.0199
CV 37%



Median 0.150
96.1% CI 0.113 to 0.221

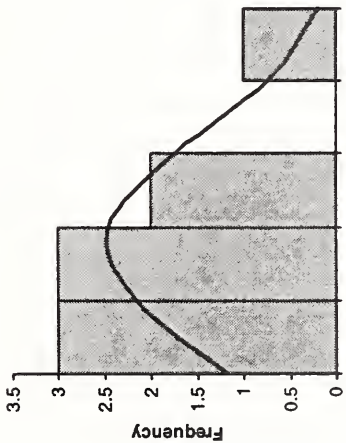
Range 0.19
IQR 0.05



	Coefficient	p
Shapiro-Wilk	0.8750	0.1391
Skewness	1.3666	0.0580
Kurtosis	1.6433	-

Percentile	
2.5th	-
25th	0.117
50th	0.150
75th	0.170
97.5th	-

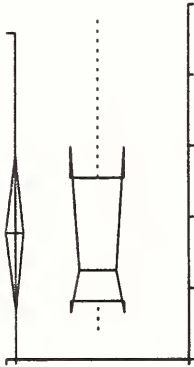
SRM 1941b
BDE 153 Results



n 9 (cases excluded: 3 due to missing values)

Mean 0.094
95% CI 0.066 to 0.122

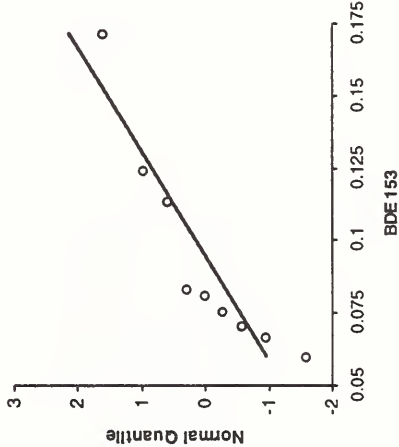
Variance 0.0013
SD 0.0360
SE 0.0120
CV 38%



Median 0.081
96.1% CI 0.067 to 0.124

Range 0.11
IQR 0.04

Percentile
2.5th -
25th 0.070
50th 0.081
75th 0.114
97.5th -



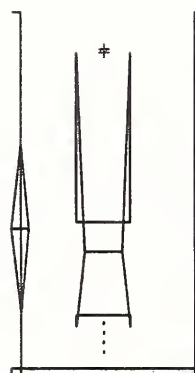
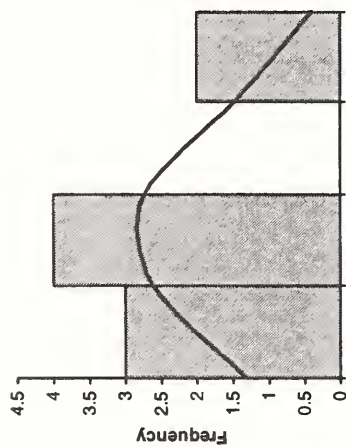
	Coefficient	p
Shapiro-Wilk	0.8456	0.0666
Skewness	1.4268	0.0484
Kurtosis	1.6715	-

SRM 1941b
BDE 154

n | 9 (cases excluded: 3 due to missing values)

Mean | 0.091
95% CI | 0.072 to 0.110

Variance | 0.0006
SD | 0.0248
SE | 0.0083
CV | 27%

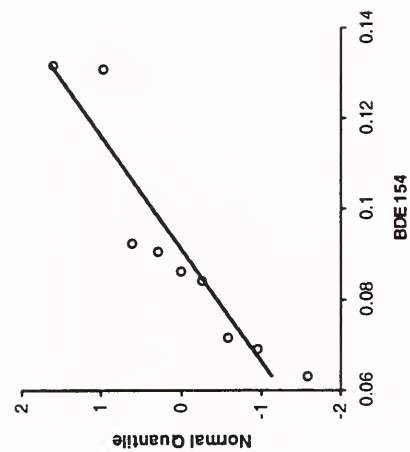


Median | 0.086
96.1% CI | 0.069 to 0.131

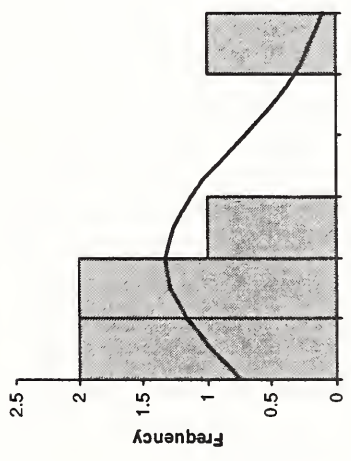
Range | 0.07
IQR | 0.02

Percentile |
2.5th | -
25th | 0.072
50th | 0.086
75th | 0.093
97.5th | -

	Coefficient	p
Shapiro-Wilk	0.8567	0.0883
Skewness	0.9363	0.1865
Kurtosis	-0.2218	-

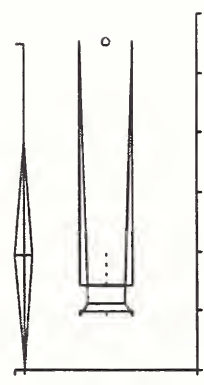


SRM 1941b
BDE 183 Results



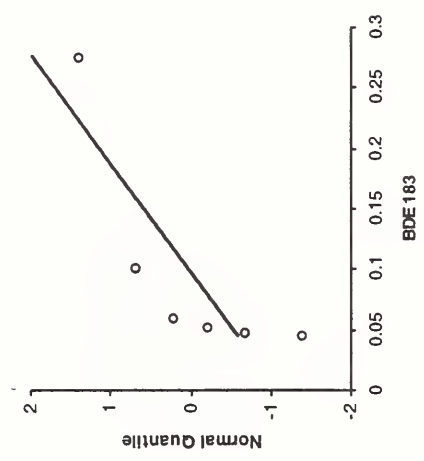
n 6 (cases excluded: 6 due to missing values)

Mean	0.097
95% CI	0.003 to 0.192
Variance	0.0081
SD	0.0901
SE	0.0368
CV	93%



Median	0.056
96.9% CI	0.045 to 0.276

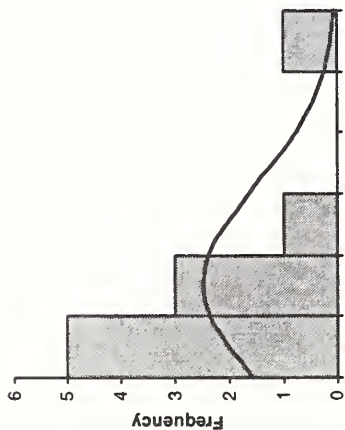
Range	0.23
IQR	0.02
Percentile	
2.5th	-
25th	0.049
50th	0.056
75th	0.070
97.5th	-



Coefficient	p
Shapiro-Wilk	0.6635
Skewness	2.1902
Kurtosis	4.8704

0.0025
-
-

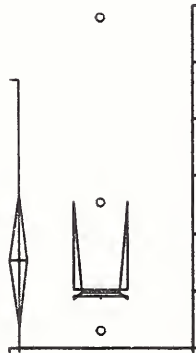
SRM 1941b
BDE 209 Results



n 10 (cases excluded: 2 due to missing values)

Mean 38.315
95% CI 9.583 to 67.047

Variance 1613.1852
SD 40.1645
SE 12.7011
CV 105%

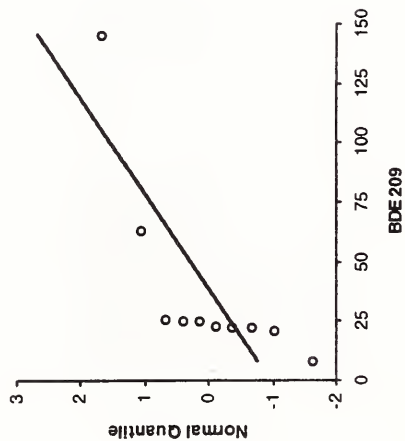


Median 24.109
97.9% CI 21.400 to 63.667

Range 137.28
IQR 2.84

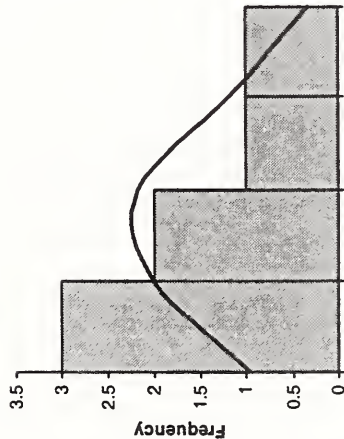
Percentile
2.5th -
25th 22.616
50th 24.109
75th 25.460
97.5th -

	Coefficient	p
Shapiro-Wilk	0.6057	<0.0001
Skewness	2.5546	0.0007
Kurtosis	6.7949	-

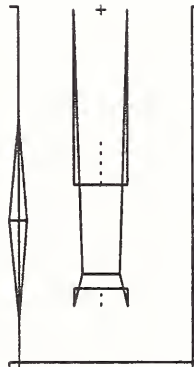


SRM 1944
BDE 28 Results

n | 7 (cases excluded: 5 due to missing values)



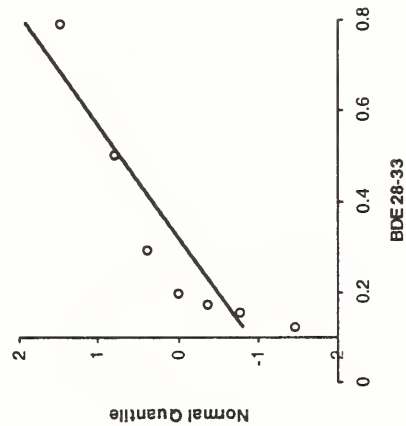
Mean	0.321
95% CI	0.096 to 0.547
Variance	0.0595
SD	0.2439
SE	0.0922
CV	76%



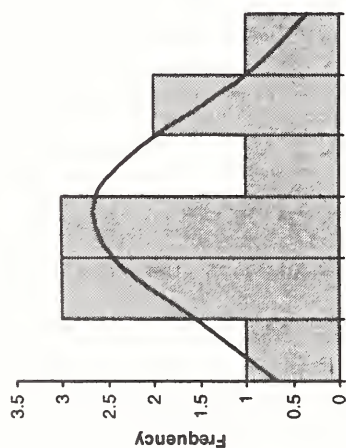
Median	0.200
98.4% CI	0.126 to 0.792

Range	0.67
IQR	0.23
Percentile	
2.5th	-
25th	0.166
50th	0.200
75th	0.400
97.5th	-

	Coefficient	p
Shapiro-Wilk	0.8078	0.0489
Skewness	1.5022	-
Kurtosis	1.5798	-



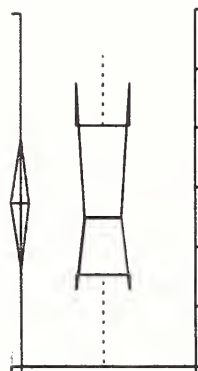
SRM 1944
BDE 47 Results



n | 11 (cases excluded: 1 due to missing values)

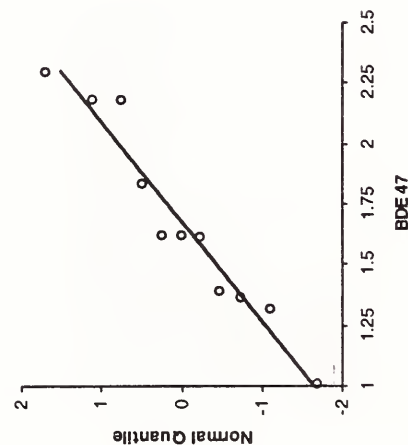
Mean | 1.679
95% CI | 1.403 to 1.954

Variance | 0.1682
SD | 0.4101
SE | 0.1237
CV | 24%



Median | 1.623
98.8% CI | 1.322 to 2.187

Range | 1.29
IQR | 0.63

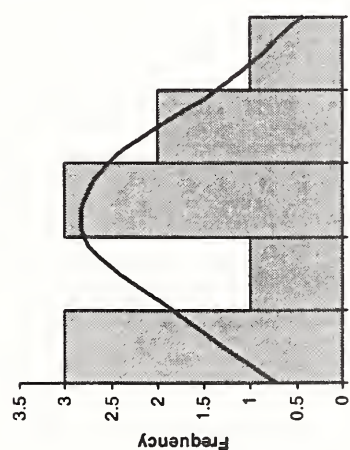


Percentile |
2.5th | -
25th | 1.381
50th | 1.623
75th | 2.010
97.5th | -

	Coefficient	p
Shapiro-Wilk	0.9371	0.4875
Skewness	0.1935	0.7602
Kurtosis	-0.8629	-

SRM 1944

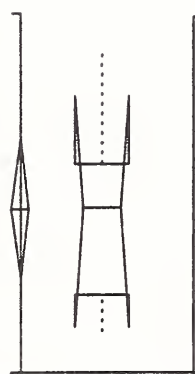
BDE 99 Results



n 10 (cases excluded: 2 due to missing values)

Mean 1.826
95% CI 1.577 to 2.075

Variance 0.1213
SD 0.3483
SE 0.1101
CV 19%

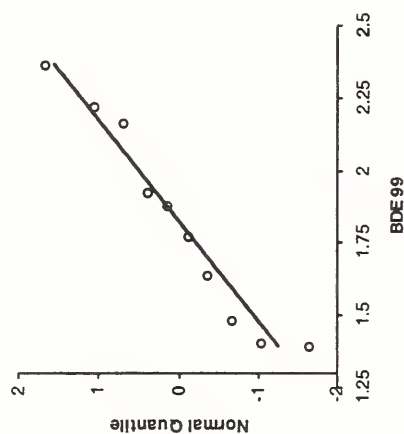


Median 1.827
97.9% CI 1.409 to 2.223

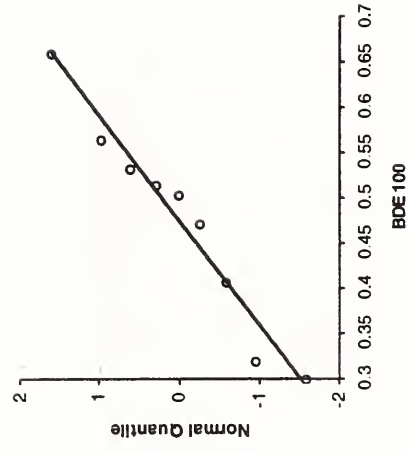
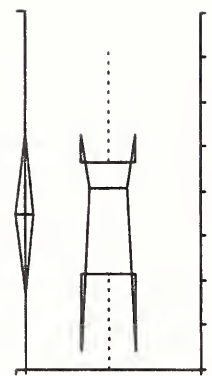
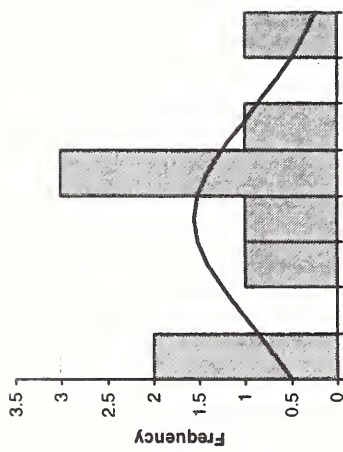
Range 0.98
IQR 0.46

Percentile
2.5th -
25th 1.525
50th 1.827
75th 1.984
97.5th -

	Coefficient	p
Shapiro-Wilk	0.9370	0.5203
Skewness	0.2016	0.7612
Kurtosis	-1.3129	-

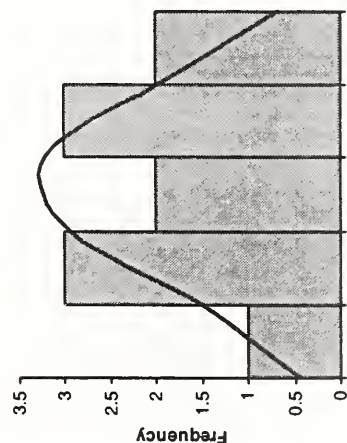


SRM 1944		BDE 100 Results		9 (cases excluded: 3 due to missing values)		
		n				
		Mean	0.475			
		95% CI	0.386 to 0.564			
		Variance	0.0134			
		SD	0.1156			
		SE	0.0385			
		CV	24%			
		Median	0.504			
		96.1% CI	0.320 to 0.564			
		Range	0.36			
		IQR	0.13			
		Percentile				
		2.5th	-			
		25th	0.407			
		50th	0.504			
		75th	0.532			
		97.5th	-			
				Coefficient	p	
				Shapiro-Wilk	0.9549	0.7436
				Skewness	-0.2151	0.7580
				Kurtosis	-0.4235	-



SRM 1944

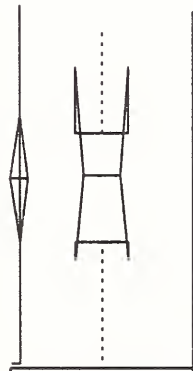
BDE 153 Results



n | 11 (cases excluded: 1 due to missing values)

Mean | 6.658
95% CI | 5.773 to 7.543

Variance | 1.7346
SD | 1.3171
SE | 0.3971
CV | 20%

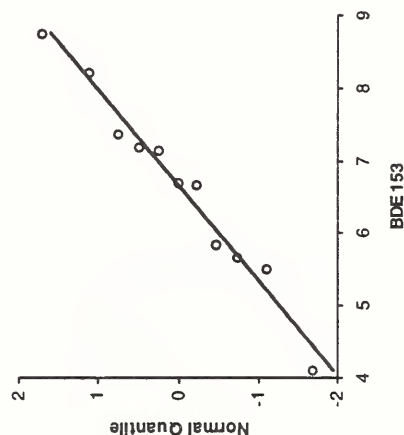


Median | 6.708
98.8% CI | 5.515 to 8.217

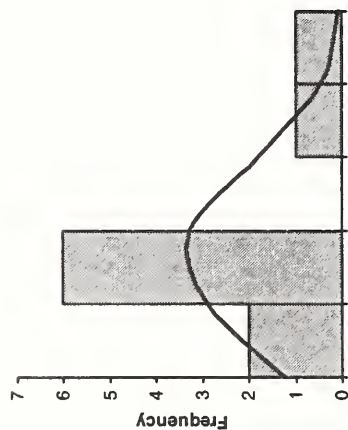
Range | 4.66
IQR | 1.53

Percentile |
2.5th | -
25th | 5.763
50th | 6.708
75th | 7.290
97.5th | -

	Coefficient	p
Shapiro-Wilk	0.9759	0.9389
Skewness	-0.3110	0.6244
Kurtosis	0.1803	-



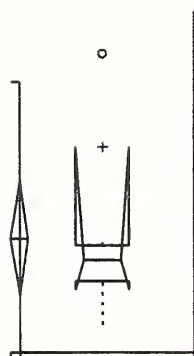
SRM 1944
BDE 154 Results



n 10 (cases excluded: 2 due to missing values)

Mean 1.335
95% CI 0.917 to 1.753

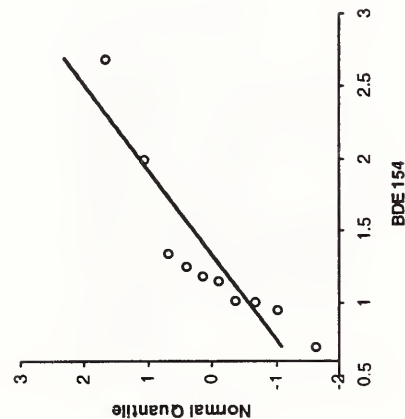
Variance 0.3414
SD 0.5843
SE 0.1848
CV 44%



Median 1.175
97.9% CI 0.957 to 2.000

	Coefficient	p
Shapiro-Wilk	0.8151	0.0221
Skewness	1.6767	0.0179
Kurtosis	2.7095	-

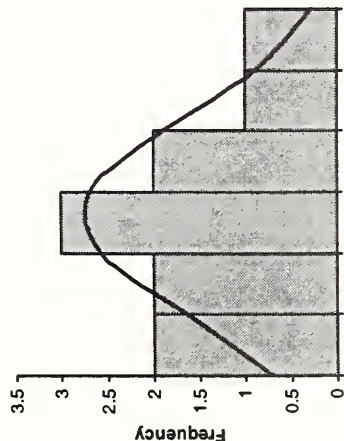
Range 1.98
IQR 0.26



Percentile
2.5th -
25th 1.017
50th 1.175
75th 1.281
97.5th -

SRM 1944

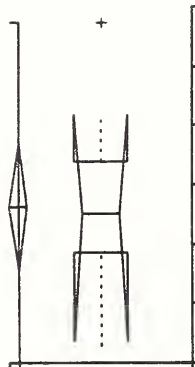
BDE 183 Results



n 11 (cases excluded: 1 due to missing values)

Mean 33.083
95% CI 27.748 to 38.419

Variance 63.0643
SD 7.9413
SE 2.3944
CV 24%

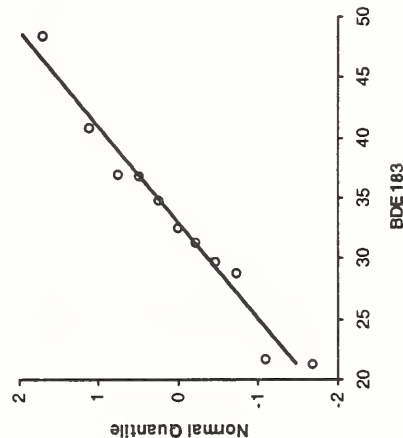


Median 32.573
98.8% CI 21.767 to 40.837

Range 27.19
IQR 7.65

Percentile
2.5th -
25th 29.284
50th 32.573
75th 36.938
97.5th -

	Coefficient	p
Shapiro-Wilk	0.9646	0.8274
Skewness	0.2807	0.6583
Kurtosis	0.3223	-

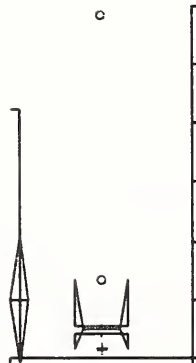
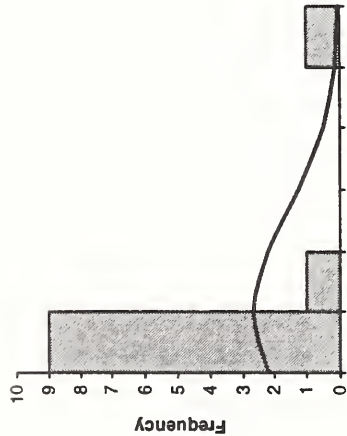


SRM 1944
BDE 209 Results

n | 11 (cases excluded: 1 due to missing values)

Mean | 250.834
95% CI | -25.279 to 526.947

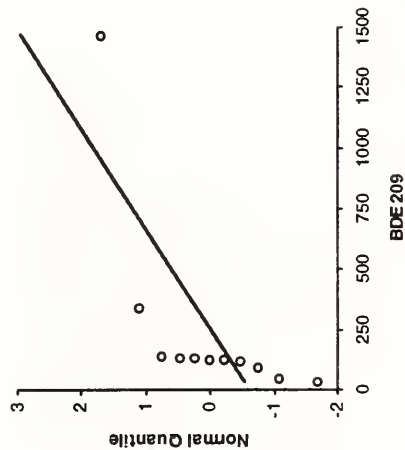
Variance | 168920.08
SD | 410.9989
SE | 123.9208
CV | 164%



Median | 127.533
98.8% CI | 44.400 to 342.000

Range | 1431.84
IQR | 32.70

Percentile |
2.5th | -
25th | 105.599
50th | 127.533
75th | 138.300
97.5th | -



	Coefficient	p
Shapiro-Wilk	0.4952	<0.0001
Skewness	3.1062	<0.0001
Kurtosis	9.9122	-

**Appendix C: List of Laboratories Participating in 2004 Inter-Comparison Exercise
on PBDEs in Sediment SRMs**

For this exercise, data were received from the following laboratories within the required timeframe. (This listing does NOT correspond to the laboratory number identification codes used in this report.)

Axys Analytical Services Ltd.
2045 Mills Rd.
Sidney, British Columbia, Canada V8L 3S8
Dale Hoover/ Coreen Hamilton

Chesapeake Biological Laboratory
1 Williams Street
P.O. Box 38
Solomons, MD 20688 U.S.A.
Susan Klosterhaus/Joel Baker

Environment Canada
Ecosystem Health Division
Environmental Conservation Branch, Ontario Region
867 Lakeshore Road, P.O. Box 5050
Burlington, Ontario, Canada L7R 4A6
Sean Backus

Environment Canada
Canada Centre for Inland Waters
867 Lakeshore Rd.
Burlington, Ontario Canada L7R 4A6
Ed Sverko

Federal Environmental Agency
Laboratory for Water Analysis, II 2.5
Bismarckplatz 1, 14193 Berlin, Germany
Peter Lepom

Indiana University
1005 E. 10th St.
Geology 541
Bloomington IN 47405, USA
Ron Hites/Lingyan Zhu

NIST – Charleston Laboratory
219 Fort Johnson Road
Charleston, SC 29412-9110 USA
Jennifer Keller

NIST-Gaithersburg
100 Bureau Drive
Mailstop 8392
Gaithersburg, MD 20899 USA
Heather Stapleton

Research Center for Eco-Environmental Sciences
Chinese Academy of Sciences
18 Shuangqing Road,
Haidian District, Beijing, China
Post Code: 100085
Guibin Jiang

Tokyo University of Agriculture & Technology
Fuchu, Tokyo 183-8509, Japan
Hideshige Takada

University of Antwerp (U.A.)
Toxicological Centre
Universiteitsplein 1
2610 Wilrijk (Antwerpen), Belgium
Adrian Covaci/Stefan Voorspoels

University of Illinois at Chicago
Environmental and Occupational Health Sciences
School of Public Health
2121 W. Taylor Street, MC 922
Chicago, IL 60612 USA
An Li

Appendix D: Methods used by participating laboratories.

<u>Lab #</u>	<u>Extraction Method</u>	<u>Extract Cleanup</u>	<u>Instrument</u>	<u>GC column</u>	<u>Internal Standard</u>
1	Ultrasonic Probe with Acetone/Hexane	activated silica gel	GC/NCI-MS	BDEs: HP 5 25 m x 0.25 mm; 0.25 µm film BDE 209: HP 1 15 m x 0.25 mm; 0.25 µm film	¹³ C BDE 209
2	Hot Soxhlet Extraction Hexane/Acetone	acidified silica and base silica	GC/NCI-MS	BDEs: HT 8 25 m x 0.22mm; 0.25 µm film; BDE 209: AT-5 12m x 0.18 mm; 0.10 µm film	BDE 77, BDE 128, ¹³ C BDE 209
3	Soxhlet Extraction Hexane/Acetone	5% deactivated and activated silica gel	GC/MS; GC/ECD	BDEs: HP 5 30 m x 0.25 mm; 0.25 µm film BDE 209: DB 5 15 m x 0.25 mm; 0.25 µm film	¹³ C BDE 139; 4'-fluoro-2,2',3,3',4,5',6,6'-nonaBDE
4	Soxhlet Extraction with DCM	acid/base silica, alumina, Florisil	GC/HRMS	DB-5 HT 30 m x 0.25 mm; 0.10 µm film	12- PBDE-15/28/47/99/100/126/153/154/183/;
5	Soxhlet with DCM/hexane	acid/bas silica gel; gel permeation chromatography	GC/HRMS	HP 5 MS 30 m x 0.25 mm; 0.25 µm film	¹³ C labeled BDE 47, 99 and 153
6	PFE with DCM	gel permeation chromatography; 5% deactivated alumina	GC/MS	BDEs: DB 5 60 m x 0.25 mm; 0.25 µm film BDE 209: DB5 15 m x 0.25 mm; 0.25 µm film	¹³ C PCB 118; ¹³ C BDE 209
7	Soxhlet Extraction with DCM	deactivated alumina and Florisil	GC/NCI-MS	DB 1 20 m x 0.10 mm; 0.10 µm film	¹³ C CDE 86; ¹³ C BDE 209
8	PFE with Toluene	gel permeation chromatography; silica gel	GC/NCI-MS	Rtx_CLPesticides 30 m x 0.25 mm; 0.25 µm film	BDE 77, BDE 140, BDE 181, ¹³ C BDE 209
9	Soxhlet Extraction Hexane/Acetone	silica gel	GC/MS	BDEs: DB 5 30 m x 0.25 mm; 0.25 µm film BDE 209: DB 5 15 m x 0.25 mm; 0.25 µm film	¹³ C BDE 118; ¹³ C BDE 209
10	24 hour Soxhlet with DCM	SPE silica cartridge eluted with hexane	GC/NCI-MS	DB-5 15 m x 0.25 mm, 0.25 µm film	¹³ C BDE 15; ¹³ C BDE 209
11	Ultrasonic Probe with Acetone/Hexane	3% deactivated silic agel	GC/NCI-MS	BDEs: DB 5 30 m x 0.25 mm; 0.25 µm film BDE 209: DB 1 15 m x 0.25 mm; 0.10 µm film	External Standard: HexaCDE
12	Soxhlet Hexane/Acetone	alumina	GC/NCI-MS	BDEs: DB 5 60 m x 0.25 mm; 0.25 µm film BDE 209: DB5 15 m x 0.25 mm; 0.25 µm film	¹³ C-CDE-156; ¹³ C-CDE-194; ¹³ C BDE 209

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2. IUPAC The International Harmonized Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories. *Pure and Applied Chemistry* **2005**, 65 (9), 2123-2144.
3. Hites, R. A. Polybrominated diphenyl ethers in the environment and in people: A meta-analysis of concentrations. *Environmental Science & Technology* **2004**, 38 (4), 945-956.
4. Keum, Y.; Li, Q. X. Reductive debromination of polybrominated diphenyl ethers by zerovalent iron. *Environmental Science & Technology* **2005**, 39 (7), 2280-2286.

